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TECHNICAL REPORT

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HYGROSCOPIC EQUILIBRIUM & TEXTURE OF FREEZE-DRIED FOODS

*Investigation of the Relationships between Moisture
Content - Water Vapor Equilibrium and
Textural Parameters in Special Freeze-Dried Foods*

by

John G. Kapsalis, Ph. D.

Chemistry Division

June 1967

Conducted under a Secretary of the Army Research and
Study Fellowship at the Swedish Institute for
Food Preservation Research (S.I.K.), Göteborg, Sweden.

UNITED STATES ARMY
NATICK LABORATORIES
Natick, Massachusetts 01760



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SUMMARY

Improved methodology for the objective evaluation of food texture was applied to the measurement of the textural parameters of special freeze-dried foods which were storage-equilibrated under different relative humidity conditions. The foods included pre-cooked freeze-dried beef, laminated freeze-dried products in the form of bite-size sandwiches, and compressed freeze-dried cubes, all of which were designed for consumption without prior rehydration. The results showed that increasing relative humidity from zero to 66% caused an increase in hardness and cohesiveness, and, in certain foods, a decrease in brittleness (crushability index). This was more pronounced at equilibrium points above the B.E.T. value for a monomolecular layer of water. Plots of physico-chemical parameters of water vapor sorption versus rheological properties at different points of the moisture sorption isotherm were examined. The selection of certain "target" relative humidity values to minimize undesirable changes upon storage, and promising avenues for continued work on the textural quality and acceptability of special freeze-dried foods are discussed.

The opinions and conclusions expressed herein are the result of independent research and study and do not necessarily reflect the views of the Department of the Army.

ACKNOWLEDGEMENTS

I express grateful appreciation to the Secretary of the Army of the United States, through his appointed Committee and Administrators of the Secretary of the Army Research and Study Fellowship, for the Award which made this work possible.

Special thanks are due to Dr. Dale H. Sieling, Dr. J. Fred Oesterling, Dr. Ferdinand P. Mehrlich, Dr. Herbert A. Hollender, Dr. J. Walter Giffey, and Mr. Kenneth H. Dobelbower of the U. S. Army Natick Laboratories, and to my able colleagues of these Laboratories, for their support or supply of experimental samples used in part of my research.

I am indebted to Professor Erik von Sydow for placing the laboratory resources of the Swedish Institute for Food Preservation Research at my disposal, and to my able colleague and friend Dr. Birger Drake for his guidance and for making available to me his previous experience on texture measurement in foods.

The construction, development of methods for graphical analysis of recorded curves, and the main part of testing of the Masticometer, as a modification of the M.I.T.-General Foods Texturometer, were done previously to this Report by Birger Drake. Certain modifications of the instrument were also carried out by Birger Drake and his co-workers at S.I.K. parallel to this work. The S.I.K. series of samples were freeze dehydrated by Nils Bengtsson and Owe Bengtsson, and the sensory evaluation was done, according to specifically designed plans, in collaboration among Birgit Johansson, Birger Drake and John G. Kapsalis. The training of the sensory panel and arrangements with the I.B.M. Data Center, Stockholm, for the statistical analysis of data were done by Birgit Johansson. Arrangements with the Goteborg Data Center on a computer program for the calculation of the Masticometer parameters were

done by Birger Drake. The chemical analysis for moisture and fat was done by (Cn.) Eriksson and his co-workers. The NIABS special foods were processed or supplied by Justin Tuomy and Mary Klicka of the U. S. Army Natick laboratories. The art work on the graphs was performed by Zoltan Benedek, and the editing work on the Report was done by Mr. Alf Erickson.

The selection of experimental variables, determination of the moisture sorption isotherms, calculation of physico-chemical variables, testing of all S.I.K. and NIABS samples by the Masticometer, measurement of masticometer curves, plotting of data, interpretation of results and writing of this Report were done by John G. Kapsalis, with laboratory assistance, in certain parts, by Zoltan Benedek, Jarl Lybeck, and Eva Örnskär.

I owe special thanks to my wife Athena for burning the midnight oil with me in planimeter work on more than 3,000 experimental curves, and for proof reading the manuscript of this Report.

I wish to thank my many and genuine Swedish friends, especially Fredrik Schoultz and Karl Remi, for helping in the problems of our temporary relocation, and for making our stay in Sweden a very fine experience.

JOHN G. KAPSALIS

INTRODUCTION

Alterations in food texture are important to the military in two classes of dried products: (1) special Army foods which are to be consumed in the dry or semi-dry state; (2) foods which require reconstitution.

The first class has become increasingly important in special, unconventional warfare, since water is not immediately necessary for consumption of the food. Sensory texture problems in such products include "hardness", "crumbliness", "dryness" and excessive "swelling" during the process of mastication. A related "mechanical" texture problem is the fragmentation and pulverization of leafy or fibrous dried foods, which occur during processing, storage and transportation. These problems cause substantial logistic and monetary losses to the Armed Forces annually.

The second class includes dried foods which, upon rehydration, show undesirable deviations from their original textural quality. The dehydration of meat may result in a "rubbery", "tough" or "spongy" reconstituted product, which requires special preparation and prolonged periods for rehydration.

Experimental work has indicated that the rheological properties of dried foods are greatly affected by the moisture content-water vapor equilibrium. As the latter changes along the adsorption or desorption loop of a moisture sorption isotherm, the textural parameters of the food also change. This may be due to transition in the hydration among different sorptive groups, to passage from mono- to multi-layer formation, and vice versa, to changes in cross-linking, to colloidal aggregation, and to other reasons.

During these changes, not merely the total moisture content, but the mechanisms of sorption and the states of water on the various polar, ionic and other groups of the food are important. On this basis, one can expect both direct and indirect relationships between the moisture content-water vapor equilibrium

and rheological characteristics of the food. In spite of some work in this area, there has been no systematic investigation of these relationships in special foods which may be consumed without rehydration. In view of the unusual and strict performance requirements on dried foods in unconventional warfare, space feeding and other applications, the Food Research Program of the Army is in need of more research in this field.

The purpose of this work was to study the relationships between certain aspects of water sorption and textural properties of special Army foods which can be consumed without rehydration. In order to have a basis of comparison, instrumental measurements in most cases were applied to both the dry and the rehydrated product. The special foods may be used in their present state of development, or they may serve as prototypes for the manufacturing of other products.

REVIEW OF LITERATURE

The literature related to the subject of this Report can be classified as follows:

Category A

Studies of the mechanism of moisture sorption and diffusion in various materials, including foods.

This literature is voluminous, and reference is made to classic and current reviews and research papers, (see bibliography in ref. 51). Most of this work deals mainly with engineering and physico-chemical aspects of dehydration, or with chemical and bacteriological problems. (1, 4, 5, 7-9, 18, 22, 29, 30, 32-44, 48, 50-53, 56-61, 66-73, 75-78, 85-92, 95, 98).

Category B

Studies of the physico-chemical, histo-chemical, and rheopsychological parameters of food texture. (2, 3, 6, 10-17, 19-21, 23-28, 31, 45, 49, 65, 79-82, 84, 96, 97, 99-111).

In this category, the work of Szczesniak and her co-workers (31, 99-104) is noteworthy. This work provided the basis for much of the experience obtained by Drake and his co-workers at S.I.K., and it has benefited this Report from both the "instrumental" and the "textural parameters" points of view. Szczesniak and her co-workers classified the textural characteristics of foods into mechanical and geometrical qualities, as well as those related to the moisture and fat content of a food product. The mechanical characteristics were subdivided into the primary parameters of hardness, cohesiveness, viscosity, elasticity, and adhesiveness, and into the secondary parameters of brittleness, chewiness, and gumminess. Table 1 shows the classification of textural parameters with the corresponding popular nomenclature suggested by these authors.

Table 1. Relationship between textural parameters and popular nomenclature (cf., refs. 100, 103)

MECHANICAL CHARACTERISTICS

<u>Primary Parameters</u>	<u>Secondary Parameters</u>	<u>Popular Terms</u>
HARDNESS		Soft-Firm-Hard
COHESIVENESS	Brittleness	Crumbly-Crunchy-Brittle
	Chewiness	Tender-Chewy-Tough
	Gumminess	Short-Mealy-Pasty-Gummy
VISCOSITY		Thin-Viscous
ELASTICITY		Plastic-Elastic
ADHESIVENESS		Sticky-Tacky-Goosey

GEOMETRICAL CHARACTERISTICS

<u>Class</u>	<u>Examples</u>
PARTICLE SIZE AND SHAPE	Gritty, Grainy, Coarse, etc.
PARTICLE SHAPE & ORIENTATION	Fibrous, Cellular, Crystalline, etc.

OTHER CHARACTERISTICS

<u>Primary Parameters</u>	<u>Secondary Parameters</u>	<u>Popular Terms</u>
MOISTURE CONTENT		Dry-Moist-Wet-Watery
FAT CONTENT	Oiliness	Oily
	Greasiness	Greasy

In more recent, U.S. Government sponsored, research by the above mentioned investigators (21), certain of these parameters were applied to the objective texture evaluation of pre-cooked freeze-dried meat in the rehydrated state.

In addition to the mechanical parameters, described above, Drake (27) at the Swedish Institute for Food Preservation Research, has suggested the "crushability index" as a useful parameter in food texture evaluation. This index and the method suggested for the graphical analysis of the curve obtained by the S.I.K. Masticometer are discussed in the chapter "Methods and Materials".

The merits and limitations of instruments available for the objective measurement of meat texture have been discussed in a recent review by Szozesniak (102). In the following, a brief description will be given of two instruments which provided the basis for the methodology used in this Report.

The M.I.T. Denture Tenderometer was designed by Proctor and his students (79-81) at the Massachusetts Institute of Technology. It is an adaption of the Volodkevich's (106) apparatus, which was constructed to measure the textural properties of meat by simulating conditions of human mastication. The Tenderometer consists of a complete set of human dentures. The upper denture is attached to a mechanical masticator (Hanau articulator) and it is moved by a driving motor, whereas the lower denture is stationary. The force exerted by the chewing action is measured through two strain gauges located in the driving arm of the upper jaw. The changes in resistance due to deformations in the strain gauges are represented as a picture on the screen of a cathode ray oscilloscope. The advantages of the instrument are: simulation of the chewing motion of the human jaws (including crushing and grinding), the ability to measure several texture parameters, and the high sen-

sitivity obtained by the use of the strain gauge and the electronic circuitry. The main disadvantages include: the difficulty of maintaining samples on the teeth, the problems associated with occlusal teeth surfaces when it is necessary to measure exactly the force acting on the food surface, and the cumbersome task of recording the force penetration patterns with a camera.

The General Foods Texturometer is a modification of the M.I.T. Denture Tenderometer and, in the interpretation of the data, it utilizes Szczesniak's classification of textural parameters. The instrument includes a Hanau dental articulator (driven by a variable-speed motor), a variable-voltage power supply, a Wheatstone bridge circuit and a fast-speed recorder with balancing potentiometer. It is different from the M.I.T. instrument in that the dentures were replaced by a punch and a sample-holding plate, that the strain-gauge sensing unit was removed from the articulator arm and relocated on the stationary bottom plate (a modification which eliminated the excessive "background signal"), and that the oscilloscope was replaced by a fast-speed recorder. Several chewing speeds were provided, but the sideways motion of the M.I.T. instrument was eliminated. The punch movement is such that it first exerts a little shearing action with its edge. This is accomplished because the punch head is at an angle in relation to the platform, and in operation, first the edge, then progressively larger area, and eventually the entire surface of the punch come in contact with the food. The reverse takes place when the punch travels upwards.

The General Foods Texturometer has been applied to the objective measurement of the texture of freeze-dried rehydrated meat in work sponsored by the U.S. Government, as mentioned above (21). Parameters applicable to meat were found to be hardness, cohesiveness, elasticity, and chewiness. The first three

parameters were determined directly from the recorded curve, whereas chewiness was calculated as the product of hardness, cohesiveness, and elasticity. Highly significant correlations were found between evaluations of tenderness by a trained panel and force applied parallel to the meat grain. Szczesniak indicated a resemblance of the parameters of meat to the components of sensory tenderness recently described by Cover et al. (13-17).

On the basis of the above mentioned instruments, Drake (24, 26), constructed a modified G.F. Texturometer, the Masticometer, which was used for texture measurements in this Report. This is discussed in detail in the chapter "Methods and Materials".

As stated in the Introduction, the main purpose of this research was to study the effect of the moisture content-water vapor equilibrium on the textural characteristics of certain freeze-dried foods in the dry state. These special foods are used in space or limited-warfare feeding and they are novel items in food science. The texture and other problems of such foods were recently discussed by Hollender (46), Klicka and Hollender (54), and others (83).

Category C

Studies of basic and applied aspects of the relationships between moisture content-water vapor equilibrium and textural parameters in foods.

Most studies in this category were made as part of, or incidental to, research on other main objectives. Specifically, the literature is very poor with regard to meat products which are designed to be consumed without rehydration, since interest in such products is presently limited to military food procurement objectives.

Davis and McLaren (18), on the basis of thermodynamic relationships derived previously, calculated the partial molal free energy, net heat and net entropy changes of water, accompanying the sorption of water vapor by eight different proteins. They found remarkable differences from protein to protein, especially between water-soluble and water insoluble proteins. Furthermore, a positive entropy change was observed for certain proteins at equilibrium points between zero and low relative humidities. This was attributed to configurational changes of the sorbing surface, which lead to local solubilization and, therefore, to an increase in randomness. In practice, such changes may appreciably affect the rheological characteristics of the system. King (53) experimenting with keratin, reported a continuous reduction in relative rigidity (determined by torsional oscillation) and an increase in the dielectric constant, as the moisture content increased from zero to 15 percent. He attributed both effects to increased rotation of polar groups in the polypeptide chain.

A great deal of research which may be related to the problems of this Report, has been conducted on fresh meat or on freeze-dried meat in the rehydrated state.

The probable mechanisms of water sorption in the fresh meat have been reviewed extensively by Hamm (39). The hydration of meat by "free" water, as expressed by the "water holding capacity" (W.H.C.), is closely related to textural characteristics. The minimum W.H.C. coincides with the maximum of muscle rigidity. During post-mortem changes, the minimum of meat hydration (rigor mortis), corresponds to a minimum of tenderness. Tenderization of meat by aging, or by application of enzymes, results in an increase of its W.H.C.

As with fresh meat, the decrease of tenderness of

freeze-dried rehydrated meat seems to be invariably associated with losses in "true" water binding capacity. This capacity depends on the ability of the muscle proteins, especially of the actin and myosin, to form a network gel, on which water is "firmly bound". Some of the viscoelastic characteristics of the muscle, e.g. elastic modulus, are determined by the state of these proteins. Therefore an associative, if not causative, relationship exists between the mechanism of water sorption on the myofibrillar proteins, and the textural characteristics of the rehydrated meat.

The moisture sorption isotherm of a food product represents a basic description of water binding relationships. Figure 1 shows a typical S-shaped food isotherm which was originally prepared for the purpose of showing main types of quality deterioration corresponding to different areas of the isotherm.

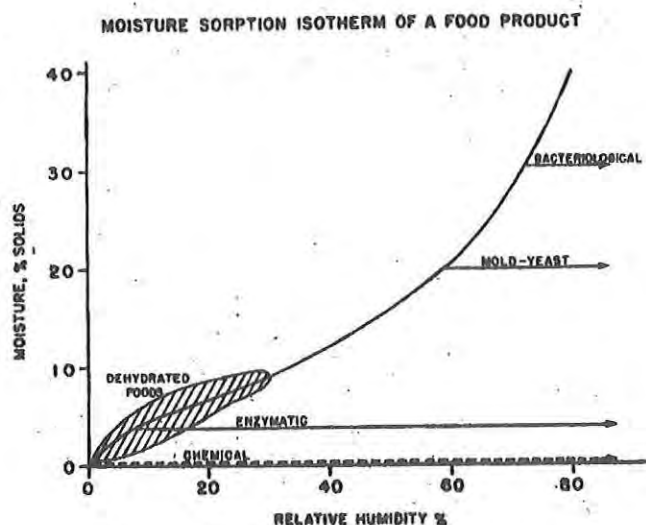


Fig. 1. Moisture sorption isotherm of a food product at 20°C.

Between zero and about 15% R.H., the curve rises steeply and it is concave to the abscissa. According to the theory of Brunauer, Emmett and Teller, (3), regarding multi-molecular adsorption, this segment represents a statistical monomolecular layer of "bound" water. The 'immobility' of water in this region has been confirmed by nuclear magnetic resonance studies on starch (93). The B.E.T. monomolecular layer is not a continuous film, but represents water molecules which are adsorbed mainly on polar sites of the food. Between approximately 15% and 50% R.H., the curve ascends more gradually, and it corresponds to the formation of a second layer of water. At still higher relative humidities, the isotherm rises steeply, it is convex to the abscissa, and it represents successive multi-layer formation. The absence of sharp discontinuities between the above segments indicated the continuous overlapping of different kinds of water binding along the isotherm. Olcott and Fraenkel-Conrat (75) showed that the amino groups play a more important role in water binding than do carboxyl groups. At a very low R.H. (about 6%), one molecule of water is bound by two amino groups. At higher R.H., (about 60%), one amino group binds $2\frac{1}{2}$ molecules of water, and the amino groups are thus saturated. At still higher R.H., uptake of water occurs mainly by "capillary condensation".

Dehydrated special foods which are to be consumed in the dry state have a moisture content which does not usually exceed the value of two B.E.T. monomolecular layers of water. Compared to its importance in fresh meat, or in meat which had been freeze-dried and then rehydrated, the W.H.C. of these special foods may be a less useful index of textural quality. Instead, the mode of water binding on polar sites of proteins and carbohydrates within the first two B.E.T. monolayers, and the extent of cross-linking between adjacent polymer chains may be the predominant factors in "crumbliness", "dryness",

"toughness" and other textural characteristics.

In most food products, the applicability of the B.E.T. equation is usually limited to relative humidities below about 0.50. This is because the B.E.T. equation contains inherent assumptions of constant number of sorption sites over all vapor pressures, and of minor lateral interactions (van der Waals).

The sorption of water on food substances is a special case of polar vapors sorbed on swelling gels. A "swelling gel" isotherm derived recently by Fugassi and his co-workers (32-34, (3)) takes this phenomenon into account. Data of Bull (9) on the sorption of water on various proteins, which fitted the B.E.T. equation only to relative vapor pressures of 0.40 to 0.50, were shown to fit the Fugassi isotherm to a relative vapor pressure of 0.90 or greater (58).

In dehydrated foods, Salwin et al., (88-90) and Kapsalis et al., (50) found close relationships between different types of chemical deterioration and the moisture content-water vapor equilibrium. In certain foods, where lipid doublebond type of oxidation is important, the presence of a monomolecular layer of water may be protective against attack by oxygen. Recently Karel (52), experimenting on highly purified model systems, elucidated important aspect of the protective role of water against lipid oxidation. In other foods, where other types of chemical deterioration predominate (ex. amino-carbonyl browning, protein oxidation, etc.), water may accelerate the deleterious change, and therefore essentially complete dehydration is necessary.

Recent work by Kapsalis et al. (51) indicated that not only the chemical stability but also the textural quality of the food may be greatly affected by the moisture content-water vapor equilibrium along the isotherm. Differences in the mode and energy of sorption between air- and freeze-dried beef were interpreted in terms of basic structural characteristics.

The textural deterioration of dehydrated foods is a spe-

cial case of chemical deterioration in its relationship to physical aspects of structure on the molecular level. Certain problems of this relationship are examined in this Report.

METHODS AND MATERIALS

I. The S.I.K. MASTICOMETER

Description

The main features and operation of the S.I.K. Mastico-meter have been briefly described previously (24, 28, 111). The instrument was developed by Drake as a modification of the M.I.T. - G.F. Texturometer, and it is shown in Figs. 2, 3, and 4. It consists of the following main parts, which are connected according to the block and the detailed diagrams in Figs. 5, 5a, 6 and 7.

a) Masticometer

Stand

Motor with gear box (30 r.p.m.), 3-phase 220 V AC

Eccenter disk with connecting rod, stroke rod, and a series of interchangeable punches

Double beam and bottom plate

Metal reed with strain gauges

Arrangement for indicating the position of the punch

b) Strain Gauge Bridge

Peekel Strain Gauge Bridge 540 DNH

c) Attenuator

Three precision decade resistances, x 10 000, x 100 and x 10 ohms

Battery circuit for the indication of the position of the eccenter disk

d) Recorder

Speedomax G, 1/4 second full deflection.

The motion transfer assembly of the apparatus is shown in Fig. 7. It includes a rotating eccentric disk which is driven by a 3-phase asynchronous motor at a speed of 30 r.p.m. The force is transmitted to the sample through a connecting rod, (which is mounted on vertical thrust bearings) a stroke rod and a punch. The eccentricity can be adjusted to any value between zero and 30.0 mm by varying the radius of rotation of the connecting rod on the circular disk. The exact setting is read on a graduated scale, parallel to the diameter of the disk, and on a corresponding vernier scale. A series of interchangeable punches ranging in diameter from 2.5 to 40 mm are available for different types of foods. In this Report, the punch diameter used was 7.5 mm for all meat samples, and 5.0 mm for the special type foods. The upper part of the punch fits into the lower section of the stroke rod by means of threaded section counterparts. A locking collar fastens the punch at any position of the threaded section, and this makes it possible to vary the minimum distance of the punch from the bottom plate and, therefore, to select the depth of sample penetration as desired.

The force receiving part of the apparatus consists of the bottom plate, the double beam, the reed and the strain gauges (Fig. 3).

The bottom plate is interchangeable, and it is threaded tightly on the center of a rectangular metal link which connects the two arms of the beam. This facilitates the use of dish-like receptacles as bottom plates, when materials of easy-flow characteristics are to be tested. (The rheological parameters of mashed potatoes and other viscous foods have been successfully tested by the Masticometer).

The beam consists of two iron arms which on the one end are bolted on the platform of the apparatus through two pairs of metal cross bars; and on the other end, as said above, the two arms are connected by the rectangular metal link which supports

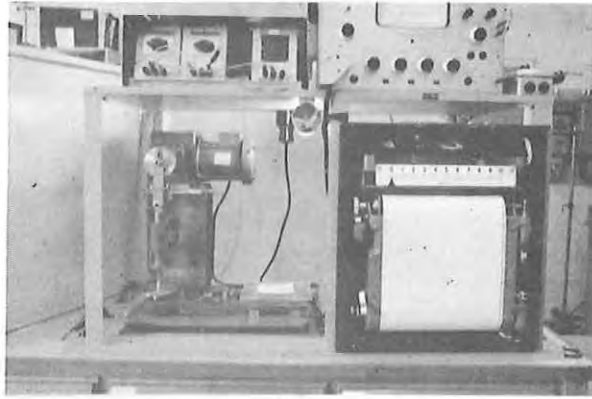


Fig. 2. Overall view of the S.I.K. Masticometer.

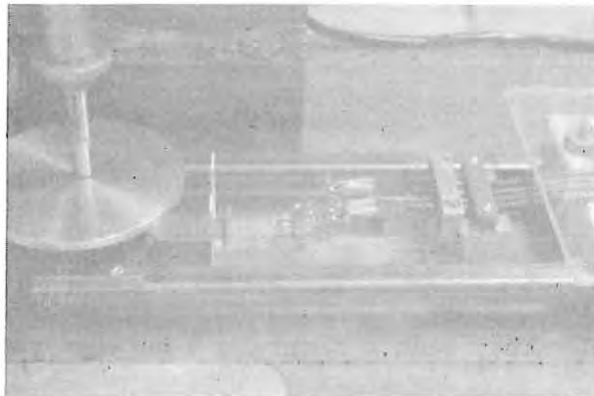


Fig. 3. Punch, bottom plate and strain gauge assembly of the S.I.K. Masticometer.

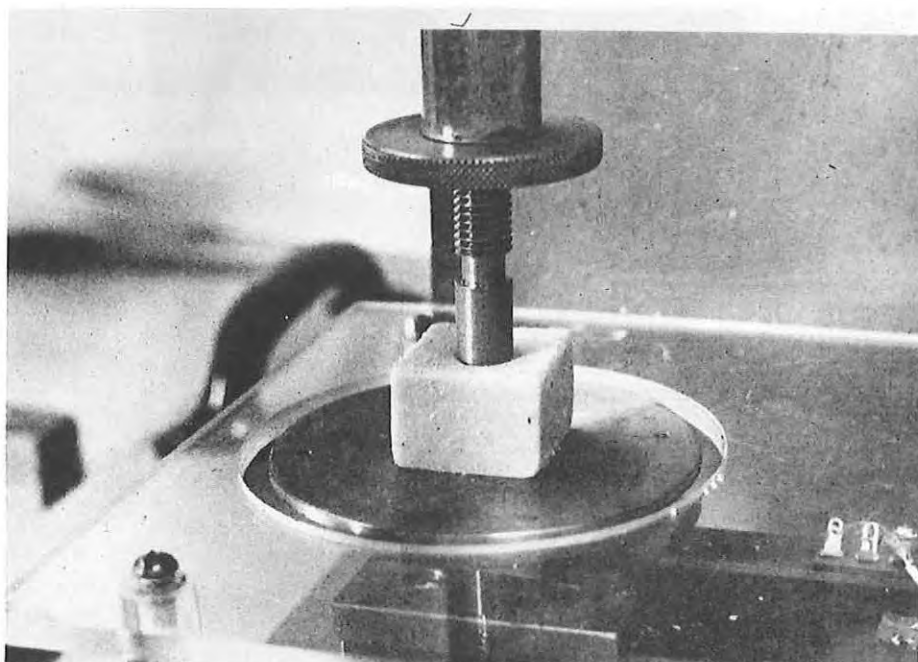


Fig. 4. Photo of rubber sample compressed between punch and bottom plate.

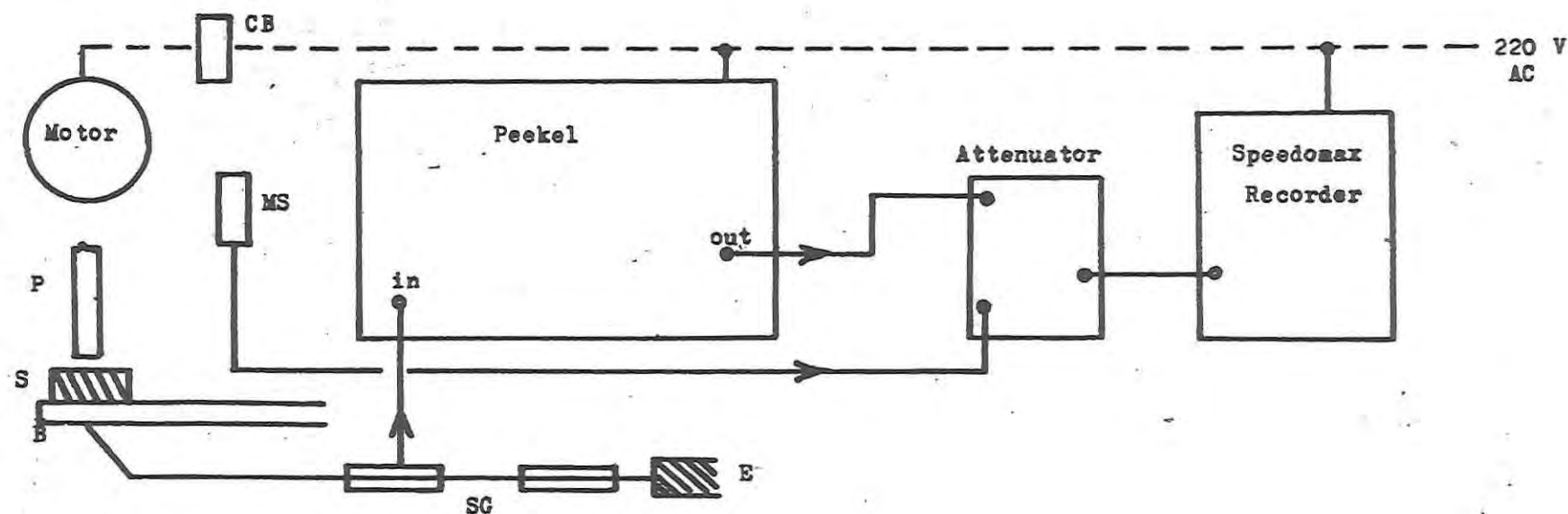


Fig. 5. Block diagram of the S.I.K. Masticometer

- P = Punch
- S = Sample
- B = Beam
- SG = Strain gauges
- MS = Microswitch for indicating the uppermost position of punch
- CB = Circuit breaker
- E = Support

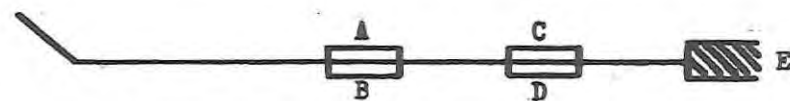


Fig. 5a. Arrangement of strain gauges

on metal reed

- A-D = Strain gauges
- E = Support

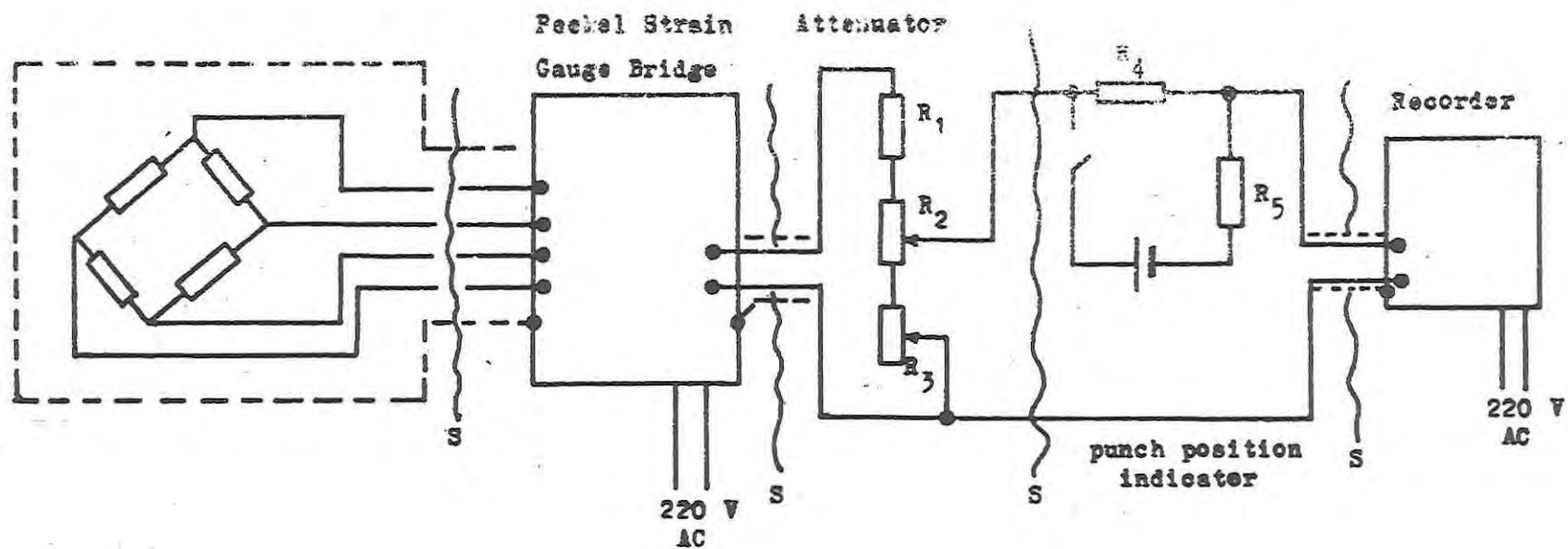


Fig. 6. Detailed circuit diagram of the measuring system of the S.I.K. Masticometer

- MS - Microswitch
- BA - Battery (1.5 V)
- R_1 - (usually) 10 kilo-Ohm
- R_2 - 10x100 Ohm
- R_3 - 10x10 Ohm
- R_4 - 10 Ohm
- R_5 - 22 kilo-Ohm
- S - Shielding

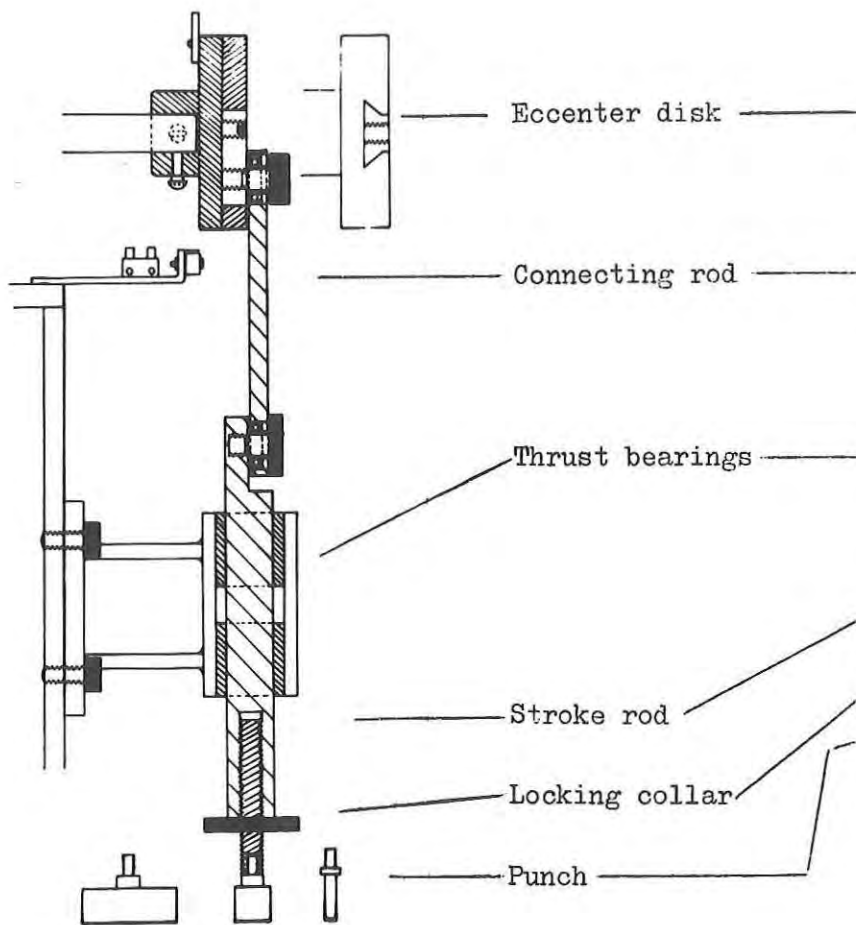


Fig. 7. The motion transfer assembly of the S.I.K. Masticometer.

the bottom plate. The underside of the link carries a hook for the suspension of weights, a step which is necessary in the process of calibrating the relative instrument force in terms of kilograms.

The reed is the part of the apparatus which bears the strain gauge. It is a 15 mm - wide metal strip whose one end bends upward and touches the underside of the rectangular metal link of the beam.

The theory and applications of strain gauges have been described in the literature (55). In principle, their operation depends on the property of electric conductors to undergo changes in resistance when subjected to mechanical deformation. In practice, a thin constantan wire is cemented to a thin plastic carrier, which in turn is cemented to the reed with the aid of a special cement. Four such strain gauges are used in the Masticometer, as shown in Figs. 5a and 6. The pressing of the sample by the punch causes a deflection of the beam and reed, and these movements are transferred through the layer of cement and the carrier to the wire of the gauges, causing variations in resistance. The latter are measured by means of a Peekel strain gauge apparatus, type 540 DNH. The imbalance in the bridge, caused by the pressure of the punch on the sample, results in a meter deflection of the Peekel strain gauge apparatus which can be read visually. In the case of the Masticometer, the output of the Peekel apparatus was transferred through an attenuator coupling to a Speedomax quick response recorder.

The relative instrument force KR exerted on the material by the punch is found by multiplying the height in mm of the recorder pen deflection A by a Factor F:

$$KR = A \times F$$

The factor F is found by dividing the setting of the micro-strain by that of the attenuator:

$$F = \text{microstrain/attenuator}$$

Modifications to the M.I.T.-General Foods Texturometer

The modifications to the M.I.T.-General Foods instrument which were incorporated into the Nasticometer include the following:

1) Substitution of a vertically moving punch for the circularly moving arm. From a physical standpoint, this resulted in better reproducibility of measurements, and it made the analysis and interpretation of the curve described by the sinusoidal movement of the punch less complicated. The curved movement of previous instruments adds new difficulties to a host of already existing variables. This includes the angle between the arm and any reference line, and the angle between the punch and the food surface; both of these angles vary continuously during testing. For these reasons, it is difficult to determine the exact depth of sample penetration, either manually or by graphical analysis of the curve. It was felt that the advantages of a vertical punch movement outweighed any possible disadvantages, incurred by the sacrifice of certain aspects in the simulated motion of the human jaw during mastication.

2) The possibility of knowing the exact point on the recorded curve where the punch was at its highest position. This point is recorded automatically by means of a microswitch, which is actuated by a pointer fixed at an adjustable position on the rotating eccentric disk. When this pointer comes in contact with the microswitch, a small voltage from a dry battery is superimposed on the attenuator, and in this way it appears as a small "kick" on the recorded curve, usually on a part of the base line (cf., Fig. 11, points p). On the basis of this mark, and knowing

the speed of the motor and of the recorder chart, it is easy to find the lowest position of the punch on the curve. As it will be shown later, this information provides the basis for the calculation of a parameter corresponding to the "recovered work", and for the interpretation of the curve in terms of "crushing", "elastic" and "total" texture behavior of the food sample.

3) The use of four strain gauges, instead of the two employed in the G.F. model. This resulted in double imbalance voltage output from the bridge to the Peekel strain gauge apparatus, and therefore, in higher sensitivity for a given deformation of the strain gauges.

Depth of sample penetration

In much of the food texture work reported in the literature, results are expressed as maximum force or other measurement at a single depth of sample penetration. Previous experience at S.I.K., indicated that single measurements may give only a partial picture of the rheological properties of the food, since many of these properties depend closely and in a nonuniform manner on the depth of sample penetration. In a similar way, during the natural process of mastication, the sensory perception of hardness and other parameters also varies with depth. It is, therefore, advantageous to know the value of textural parameters at any point within the path of a chewing cycle. Once this is known, a definite depth can be selected for further measurements, in order to obtain meaningful and comparable data. The Masticometer provides for both manual setting of the depth of sample penetration, and for the exact estimation of sample thickness and of penetration depth on the basis of a graphical analysis of the curve.

Manual setting and corrections of the depth of sample penetration

The depth of penetration of the punch into a food sample is set by adjusting the distance of the punch from the bottom plate, when the punch is placed in its lowest position. This is done by

means of the threaded sections of the punch and stroke rod, as explained previously. The desired punch-to-bottom-plate distance can be accurately set on the basis of a series of standard thickness steel plates. This distance when subtracted from the thickness of the sample gives the nominal penetration depth or Nom PD. However, the pressure applied to the sample by the punch causes also a downward deflection of the beam, and as a result, the distance between the punch and the bottom plate becomes larger than it would have been if the beam had been absolutely stiff. The punch travels into the food to a shorter distance than the Nom PD indicates, and a correction ΔX is necessary in order to find the real penetration depth. Such a correction can easily be calculated, since the deflection of the pen recorder, after multiplication by the factor F, gives a measure of the depression of the beam.

Assume, for example, that a 1.00 mm sheet of steel on the bottom plate gives D mm pen deflection, at microstrain setting S and attenuator setting A. This gives the instrument force KR for one millimeter beam deflection as:

$$KR/mm = D \cdot (S/A)$$

and a factor

$$f = \frac{A}{DS} \text{ mm/KR}$$

For experiments mentioned in this report, the value obtained by the last equation in a large number of replicate measurements was $0.614 \times 10^{-3} \text{ mm/KR}$. The correction ΔX in mm is found by multiplying the force KR by the factor f.

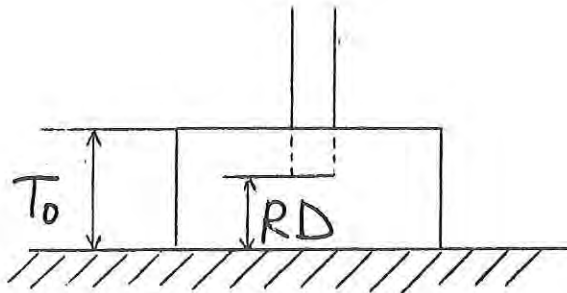
$$\Delta X = f \cdot KR \text{ mm}$$

Consequently, the real penetration depth PD is:

$$PD = \text{Nom PD} - \Delta X \text{ mm}$$

In the Masticometer, the deflection of the beam does not usually exceed 1.5 mm, within the range of hardness encountered in most foods, and when not too large PD values are employed. Most samples tested in this report caused a deflection of less than 1.00 mm. Exceptions to this were shown by certain special foods (strawberry cubes, melba toast and beef bites). Probably because of long storage, these samples were so hard that as little as 1.00 mm nominal penetration depth caused a too high deflection of the beam. The same foods were impossible to chew by a sensory panel, and they were excluded from texture measurements.

Graphical estimation of sample thickness and of penetration depth



According to the previous discussion, the percent nominal penetration depth (% Nom PD) can be calculated on the basis of the sample thickness T_0 and of the remaining depth RD:

$$\% \text{ Nom PD} = \frac{T_0 - RD}{T_0} \times 100$$

Of these quantities, RD is known exactly from the minimum

distance of the punch from the bottom plate. The thickness T_0 , however, is difficult to measure accurately by mechanical means, especially on meat and other food samples which have irregular surfaces. A method for the exact calculation of sample thickness by graphical analysis of the curve, described by the movement of the punch during penetration, was developed by Drake at S.I.K. According to this method, the position of the punch at any time during operation can be calculated on the basis of Fig. 8.

The outer circle represents the circumference of the eccenter disk, and the inner circle the positions of the end of the connecting rod VE, during one revolution; this end is fixed to a point on the eccenter disk as the latter rotates during operation. The distance AF is the eccentricity ECC; line AF makes an angle ϕ with vertical line DB. As the eccenter disk rotates, angle ϕ is 0° at the uppermost position of the punch, and it completes 360° at the end of a full rotation of the disk. At the uppermost position, the top and bottom ends of the connecting rod are at D and O, respectively. Upon rotation, when point D reaches A, point O reaches B, and the vertical distance travelled by the plunger is Δ .

From triangles ABC and AFC it is easily seen that:

$$(VE)^2 = (AC)^2 + (CB)^2 = (ECC \cdot \sin \phi)^2 + y^2$$

$$y = \sqrt{(VE)^2 - (ECC \cdot \sin \phi)^2}$$

$$\Delta + VE = y + (ECC - ECC \cdot \cos \phi)$$

$$\Delta = y + (ECC - ECC \cdot \cos \phi) - VE$$

$$\Delta = \sqrt{(VE)^2 - (ECC \cdot \sin \phi)^2} + ECC (1 - \cos \phi) - VE$$

In the last equation the distance Δ is given as a function of

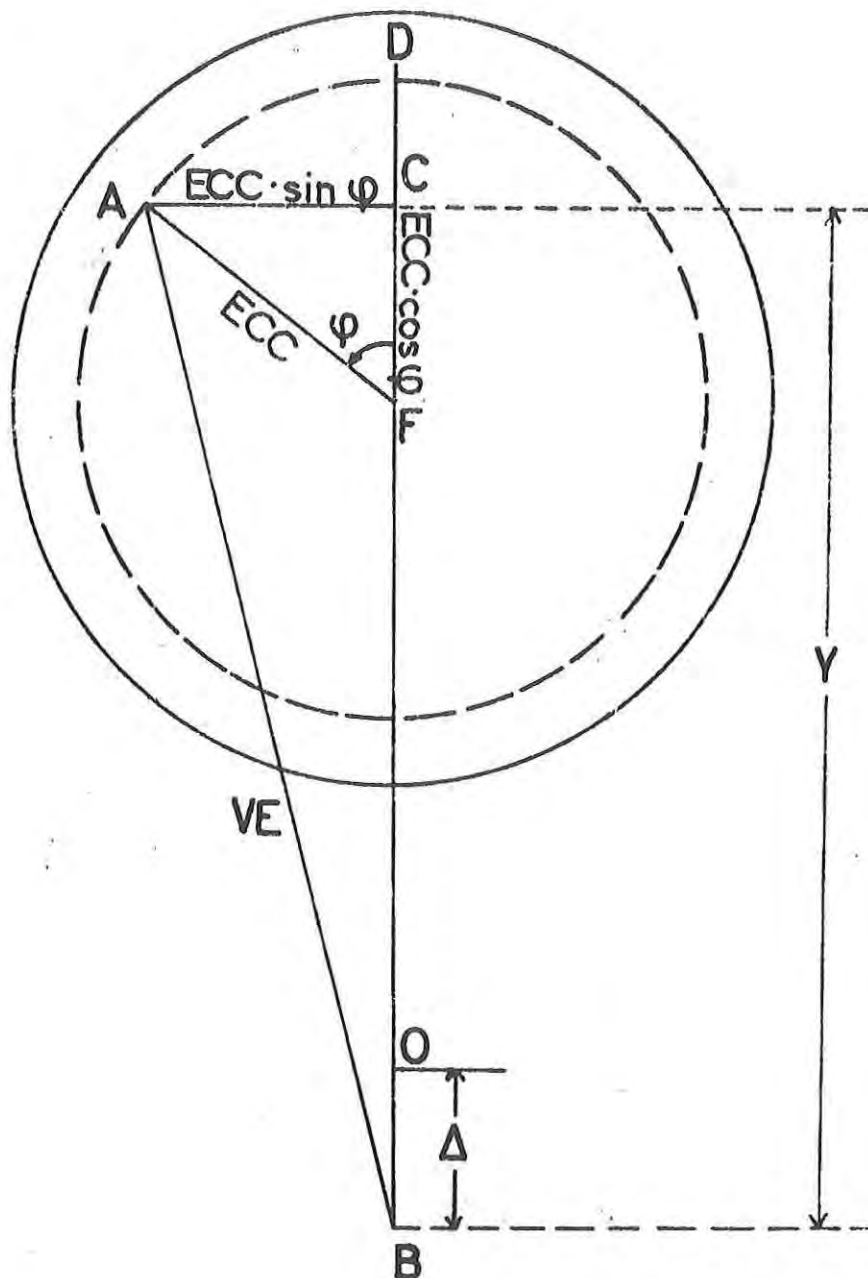


Fig. 8. Diagram for the calculation of the height of the punch as a function of time.

ECC = eccentricity

D, O = uppermost position of the ends of the connecting rod VE

A, B = position of the ends of the connecting rod VE at time t.

Δ = vertical distance travelled by the lower end of the connecting rod VE in time t.

the angle ϕ . Table 2 presents values of the maximum distance of the punch from its lowest position, calculated for different angles ϕ and at different eccentricities. Fig. 9, bottom curve, shows the plotting of these data for ECC = 10 mm on a 4:1 scale. It is seen that a nearly sinusoidal curve results. On the top of Fig. 9 is an experimental curve obtained with pre-cooked freeze-dried beef in the rehydrated state. Point b_1 on the bottom curve corresponds to the uppermost position of the punch before the first experimental peak. This position is registered automatically on the recorder curve in the form of a small peak p , as explained previously in this Report. Point a_1 is the lowest position of the punch, and point b_2 again the uppermost position, before the recording of the second experimental peak. As the plunger moves downward, it makes contact with the food at a point on the upper curve t_1 which corresponds to the point t_2 on the bottom curve. Therefore, on the basis of this graphical analysis, the distance T_0 which corresponds to the thickness of the food, can be accurately and conveniently measured from the scale on the ordinate.

Another way, suggested by Drake, for the measurement of sample thickness depends on the relationship between the length of the base line l and the nominal penetration depth Nom PD. When Nom PD = 0 (zero) $l = 100$ mm = maximum, at the present speed of the recorder chart (100 mm/sec.) and of the Masticometer motor (30 r.p.m.). When Nom PD = ECC, $l =$ (almost exactly) 50 mm, and when Nom PD = 2 ECC, $l = 0$. For intermediate values, a definite relationship exists between Nom PD/ECC and l , which can be quantitatively determined from the plotting of these two variables when materials of known thickness are used. The standard curve thus obtained is valid for any eccentricity ECC.

In the present Report, a standard curve of Nom PD/ECC vs. l was established by using a series of samples of erasing rubbers. The thickness of these rubbers was measured to 0.005 mm with a micrometer in 4 replicates for each penetration. The results are

Table 2. Values of maximum distance of punch from lowest position, calculated from the graphical analysis of the Masticometer curve.

ϕ°	Distance from lowest position for mm eccentricity		
	10.0	20.0	30.0
0	0.0	mm 0.0	0.0
9	0.1	0.2	0.3
18	0.4	0.8	1.1
27	1.0	1.8	2.4
36	1.7	3.2	4.3
45	2.7	5.0	6.7
54	3.8	7.0	9.7
63	5.1	9.4	13.1
72	6.5	12.1	16.9
81	8.0	15.1	21.2
90	9.5	18.2	25.8
99	11.1	21.3	30.6
108	12.7	24.5	35.5
117	14.2	27.6	40.3
126	15.6	30.6	44.9
135	16.8	33.2	49.1
144	17.9	35.6	52.9
153	18.8	37.4	55.8
162	19.4	38.5	58.1
171	19.9	39.8	59.5
180	20.0	40.0	60.0

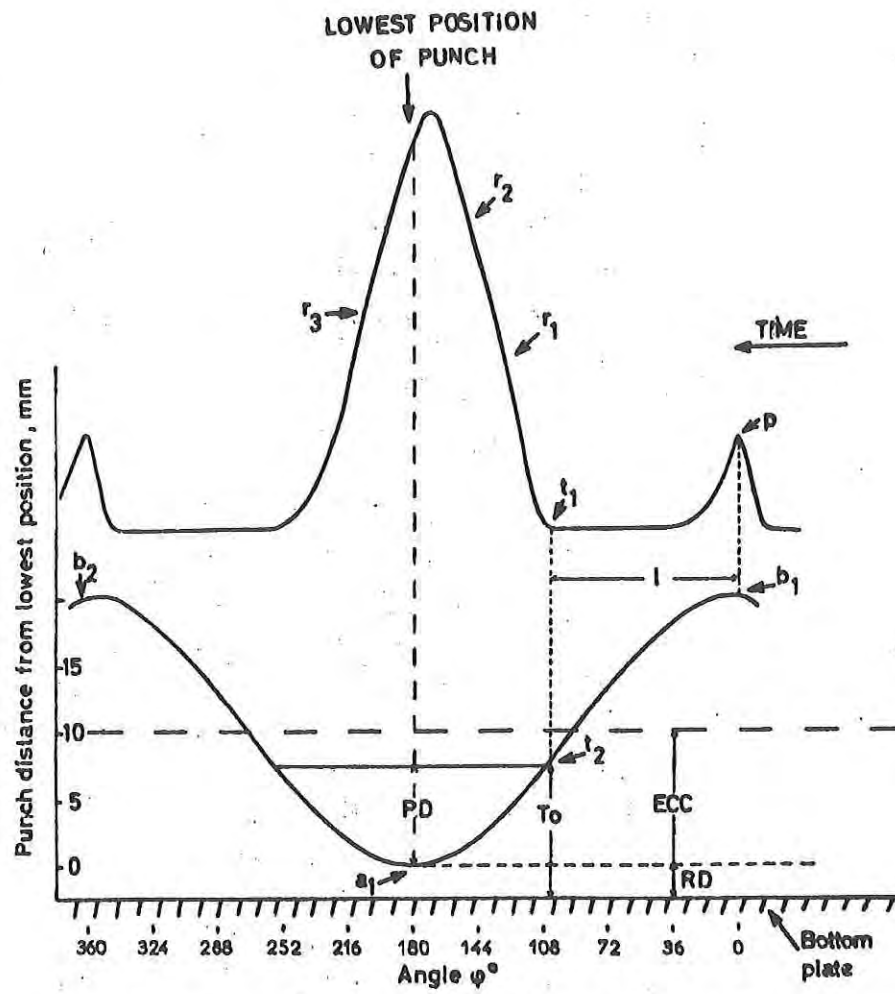


Fig. 9. Graphical estimation of sample thickness and penetration depth.

shown in Table 3 and Fig. 10. A small deviation of the standard from the theoretical curve was observed, due probably to mechanical reasons. This deviation points to the necessity of establishing a standard curve with any new instrument, as well as with any new sample holder on the bottom plate. On the basis of Fig. 10, a working table of Nom PD/ECC vs. l values was made, and this served for the calculation of the thickness of all foods, except of the special sandwich samples processed by NLABS. The thickness of these samples exceeded the maximum value covered by the standard curve, and it was measured directly with a micrometer.

Determination of food hardness at different points of a chewing cycle from a single Masticometer curve.

In addition to the above mentioned method for the calculation of sample thickness, the graphical analysis of the curve makes it possible to determine accurately the depth of punch penetration into the food at any point r_1, r_2, r_3 etc., of the chewing cycle (Fig. 9). Correspondingly, the experimental top curve may serve for the calculation of the force exerted by the punch at any point r_1, r_2, r_3 etc., on the basis of the distances of these points from the base line. As a result, a complete profile of hardness at different points within one chewing cycle can be made using a single experimental curve. This may substantially reduce the necessary number of replicate measurements for texture work. In the Denture tenderometer of Proctor et al. (79-81) a "texture profile" was represented by the characteristic loop on the oscilloscope screen. This loop also needed a certain analysis in order to express it in terms of hardness at different points of the chewing cycle. In the present Report, this application of the Masticometer punch curve was not tested on the experimental food samples. Instead, separate experimental curves were obtained in sufficient number of replicate measurements at different penetration depths. This approach was selected in view of the textural heterogeneity of meat, and of the fact that the above graphical analysis was

Table 3. Examples of the calculation of sample thickness by graphical analysis of the Masticometer curve

Material	Penetration No	Remaining depth, mm	Thickness mm	l, mm	Nom PD/ECC
Läufer 333	1	6.00	10.728	62.8	0.4715
			10.722		
			10.715		
			<u>10.697</u> 10.716		
	2	6.00	10.620	63.9	0.4620
			10.617		
			10.619		
			<u>10.624</u> 10.620		
	3	6.00	11.024	61.8	5.009
			11.003		
			11.009		
			<u>10.009</u> 10.761		
	4	3.00	10.710	53.0	0.7709
			10.713		
			10.740		
			<u>10.670</u> 10.708		
	5	3.00	10.748	53.0	0.7762
			10.758		
			10.798		
			<u>10.745</u> 10.762		
Artgum 250	1	6.00	15.536	47.5	0.9540
			15.545		
			15.545		
			<u>15.535</u> 15.540		
	2	6.00	15.559	48.0	0.9563
			15.562		
			15.569		
			<u>15.565</u> 15.564		
	3	10.5	16.019	60.5	0.5504
			16.005		
			16.000		
			<u>15.994</u> 16.004		

Table 3. continued

Material	Penetration No	Remaining depth, mm	Thickness mm	l mm	Non PD/ECC
Magic Rub	4	10.5	16.100 16.120 16.110 <u>16.120</u> 16.112	60.0	0.5612
	1	6.00	12.893 12.836 12.871 <u>12.375</u> 12.881	56.2	0.6881
	2	6.00	12.849 12.846 12.849 <u>12.850</u> 12.848	56.2	0.6848
	3	6.00	13.581 13.574 13.580 <u>13.607</u> 13.586	53.1	7.586
	4	9.00	11.226 11.227 11.231 <u>11.224</u> 11.227	73.5	0.2227
	5	9.00	11.223 11.219 11.218 <u>11.224</u> 11.221	73.0	0.2221
	1	9.00	20.168 20.165 20.169 <u>20.164</u> 20.166	44.5	1.1166
	2	9.00	20.128 20.132 20.105 <u>20.132</u> 20.124	45.6	1.1124
	3	15.00	19.655 19.677 19.681 <u>19.670</u> 19.671	68.0	0.4671
Pelican special 16					

Table 3 continued

Material	Penetration No	Remaining depth, mm	Thickness mm	l mm	Nom PD/ECC
Pink Diamond	4	15.00	19.570 19.552 19.565 <u>19.562</u> 19.562	63.5	0.4562
	1	4.50	9.783 9.789 9.775 <u>9.785</u> 9.784	61.0	0.5283
	2	4.50	10.222 10.221 10.222 <u>10.224</u> 10.222	60.0	0.5723
	3	7.50	10.545 10.545 10.544 <u>10.548</u> 10.546	69.1	0.3045
	4	7.50	10.139 10.129 10.130 <u>10.139</u> 10.134	72.0	0.2634
	1	4.50	8.758 8.753 8.756 <u>8.756</u> 8.756	64.2	0.4256
	2	6.00	8.784 8.783 8.783 <u>8.780</u> 8.782	70.6	0.2782
State Grade					

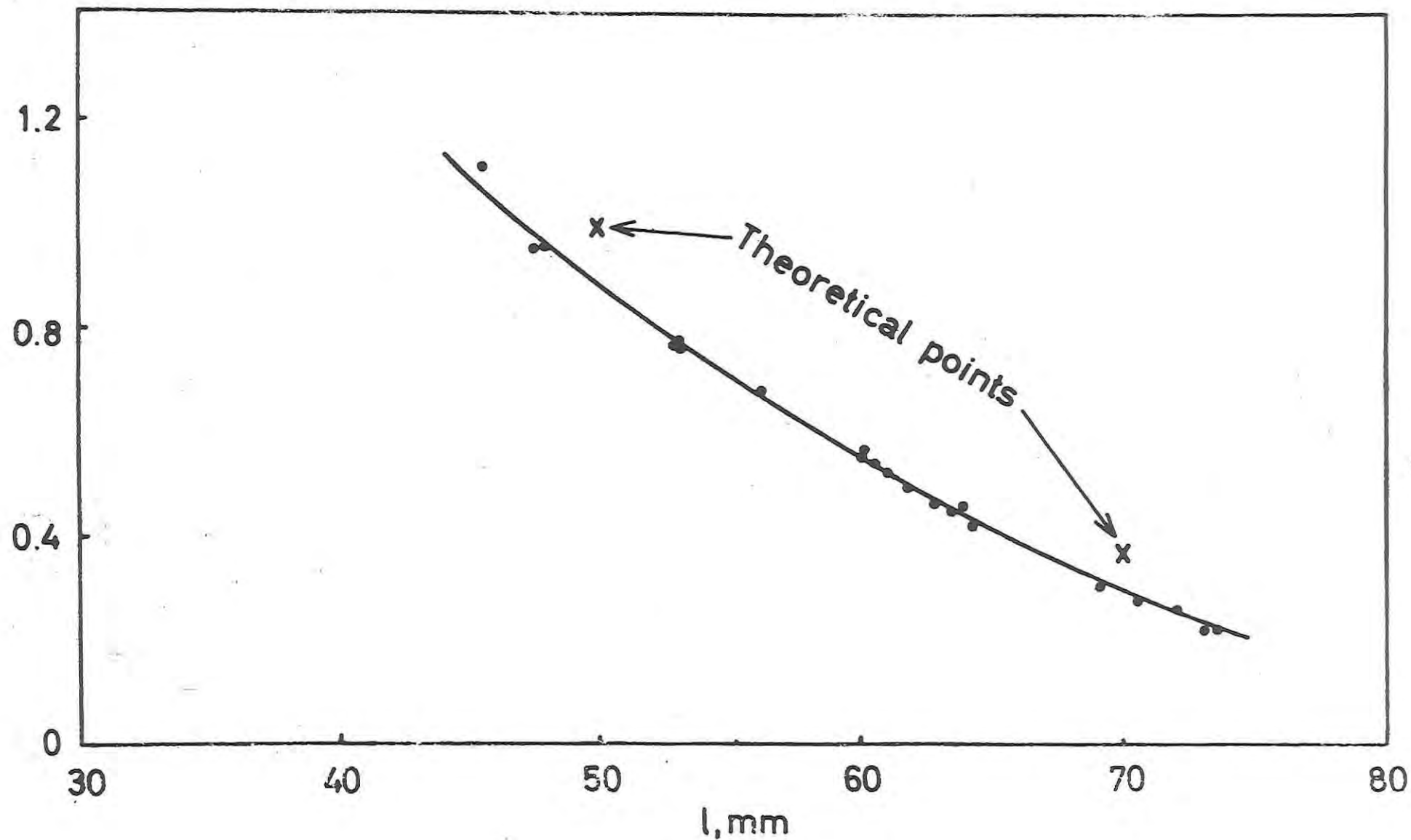


Fig. 10. Standard curve for the calculation of sample thickness and penetration depth by graphical analysis of the Masticometer curve

still under investigation while other phases of this work were underway.

Calibration of beam deflection and of instrument force in terms of kilograms

The relationship between force in kg and mm beam deflection was found by suspending a series of weights from the hook of the rectangular metal link of the beam, and measuring the deflection of the beam with a high sensitivity micrometer. A great number of such measurements gave a practically straight line relationship between force and beam deflection, with an average of 16.35 kg force per millimeter beam deflection.

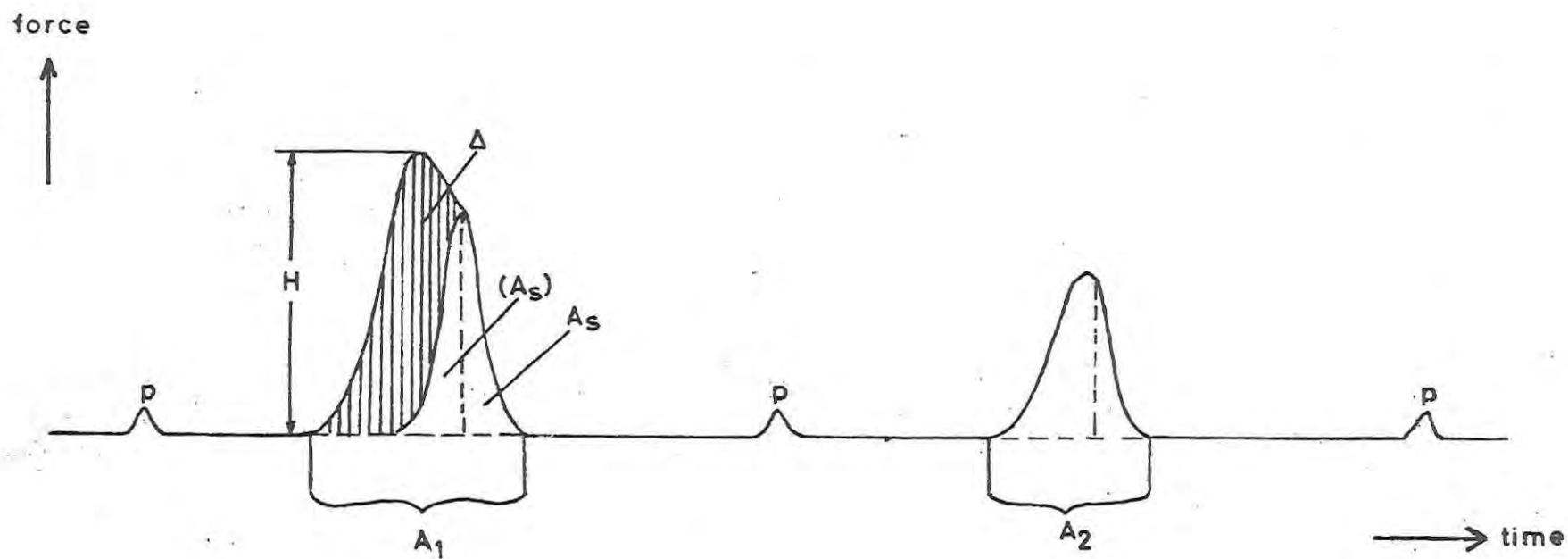
The relationship between force in kg and instrument force KR, was found by using kg weights in the same manner as above, and measuring the deflection of the recorder pen, at a certain micro-strain and attenuator setting. The results are shown in Table 4.

Textural parameters as measured by the Masticometer

Fig. 11 shows schematically two curves recorded consecutively by the Masticometer, and representing the first (area A₁) and second (area A₂) chewing cycles on the same food sample. Small peaks p represent the time when the punch was at its uppermost position, whereas the vertical dashed lines within each experimental peak represent the time when the punch was at its lowest position. In this Report, the following textural parameters, derived from the Masticometer curve, were used:

Hardness. This is the maximum force exerted by the punch on the food during penetration. The force can be expressed in relative instrument units KR for the size of punch used, or in terms of kg/cm². The KR value is found by multiplying the maximum height H in mm of the first peak A₁ by the factor F = microstrain/attenuator:

$$KR = H \times F$$



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Fig. 11. Diagram for the evaluation of hardness, cohesiveness, and "crushability index".

p = uppermost position of punch

H = height of first peak; A_1 and A_2 = total surface areas of first two peaks;

A_s = surface area of second part of first peak (i.e., the part recorded after the punch's passing of its lowest position); $\Delta = A_1 \text{ minus } 2A_s$.

The hardness HR in kg/cm^2 is found by multiplying KR by the conversion factor, 1.446×10^{-2} , and dividing the product by the cross-sectional area "a", in cm^2 , of the punch:

$$\text{HR} = \text{KR} \times 1.446 \times 10^{-2} \times a^{-1}$$

The conversion factor 1.446×10^{-2} represents the number of kg per unit KR, and it was found experimentally as in Table 4.

Cohesiveness. This is the ratio of the area under the peak A_2 to the area under peak A_1 , as measured by a planimeter. It is conveniently expressed in percent cohesiveness, % C:

$$\% C = \frac{\text{Area } A_2}{\text{Area } A_1}$$

The above mentioned parameters are among those investigated by Szczesniak (99, 100, 103, 104) in connection with the General Foods Texturometer, and they are equally applicable to the Masticometer. In addition, Drake (27) discussed a texture parameter which he called "crushability index", according to the following examination of the Masticometer curve (Fig. 11).

When the punch in its downward movement penetrates the food and reaches its lowest position, indicated by the dashed vertical line in curve A_1 , a certain amount of work is being put into the material. When the punch in its upward movement, beginning from the dashed vertical line toward the right, rises to the surface of the food, a certain amount of this work is being recovered. The area A_s corresponds to this recovered work, and represents the elastic behavior E of the food. (The words "corresponds", "represents" and "behavior" are purposely used here, because mechanical work is the product of force and distance, whereas the Masticometer area under the peak is the plot of force vs. time). If area A_s is mirrored to the left, area (A_s) results. The difference Δ between total area A_1 and $2A_s$ represents the crushing or non-elastic

Table 4. Values obtained in the calibration of the Masticometer by static loading of beam

Weight kg	Instrument setting		Factor F	Height, H on strip chart mm	Force (H x F)	I/KR	kg/KR	KR/kg
	microstrain	attenuator						
5	100	60	1.67	205.0	342	2.923×10^{-3}	1.460×10^{-2}	68.4
10	300	90	3.33	208.5	694	1.430×10^{-3}	1.440×10^{-2}	69.4
15	300	60	5.00	209.0	1045	0.975×10^{-3}	1.436×10^{-2}	69.7
20	300	50	6.00	231.8	1391	0.719×10^{-3}	1.438×10^{-2}	69.6
25	300	40	7.50	229.0	1718	0.582×10^{-3}	1.455×10^{-2}	68.7
Mean	-	-	-	-	-	-	1.446×10^{-2}	69.2

behavior of the food which expressed as the ratio of Δ to A_B is the "crushability index" CI:

$$CI = \Delta / A_B$$

In this Report, the sum of the crushing + 2x(elastic behavior) is called the "total texture behavior" Σ of the food:

$$\Sigma = \Delta + 2E$$

II. PRE-COOKED FREEZE-DRIED BEEF PROCESSED BY S.I.K.

Types of samples, and processing

As stated previously, the main purpose of this research was to study the effect of different conditions of moisture content-water vapor equilibrium on the textural properties of certain special foods, and particularly of pre-cooked freeze-dried meat. In this latter case, it was necessary to: (a) obtain a sufficient number of samples of similar textural characteristics within each group, and of different textural characteristics between groups, in order that a meaningful statistical design could be applied; (b) control the thickness of each sample, in order to insure comparable cooking treatment of all pieces and to facilitate testing by the Masticometer; and (c) control the temperature of cooking within each sample, by controlling the temperature-time variable and the dimensions of each sample. Item (c) was considered essential in view of the important effect of cooking on the textural properties of meat (105).

To satisfy these requirements, meat of the semimembranosus muscle from the left and right sides of four beef animals, of a grade corresponding to "U.S. Good", was selected and processed by the Pilot plant of S.I.K. The animals had the following characteristics:

Animal I, about two years old, meat 13.2% fat on dry basis

Animal II, about five years old, meat 16.0% fat on dry basis

Animal III, about two years old, meat 7.0% fat on dry basis

Animal IV, about five years old, meat 17.3% fat on dry basis

In order to obtain uniform thickness of the experimental samples, the following procedure was selected:

The raw meat was frozen in a freezer chamber maintained at -30°C for 17 hours. Each muscle was then cut into 16 consecutive slices of 10 mm thickness, numbered 1 to 16. Each of these slices was cut into three pieces, coded a, b and c, using the sharp end of a steel cylinder. Samples a and b were whole circular pieces of approximately 4 cm diameter, and they were used for the actual experimental work. Pieces c were the remaining fragments of the slice and were used only for the preliminary setting of the controls of the Masticometer, before testing of the pieces a and b.

The above pieces were cooked at the Experimental Kitchens of S.I.K. by immersion in water, using a water bath which was thermostatically controlled within $\pm 0.5^{\circ}\text{C}$. The temperature of the samples was measured by thermocouples embedded into the center of the meat. The cooking was stopped when the following temperatures were reached:

Meat pieces from animal I:	77°C
" " " " II:	66°C
" " " " III:	77°C
" " " " IV:	68°C

The above cooking treatments were selected in an effort to accentuate textural differences due to the age of the animals, and to therefore produce a wide range in the individual textural parameters to be measured. Samples I and II were tested only by the Masticometer; samples III and IV were tested by both the Masticometer and a specially trained sensory panel. In this latter case, the lowest cooking temperature of 66°C (samples II) was raised to 68°C (samples IV), in order to facilitate acceptance for testing by the panel.

After cooking, the meat pieces were placed in labelled aluminum receptacles, and they were refrozen in a freezer chamber maintained at -30°C for 17 hours. (Although this double freezing was not representative of normal commercial practice, it was considered necessary in order to obtain samples of uniform thickness before cooking).

The dehydration of the frozen samples was done in a "Kylteknik" laboratory model freeze drier. The plate temperature was 100°C at the beginning, and it gradually dropped to 40°C at the end of the drying period. The maximum surface temperature of the food was 40°C and the maximum ice temperature, as determined by thermocouples embedded into the frozen part of the food, was -25°C . The pressure inside the vacuum chamber was 0.1 mm Hg at the beginning, and 0.01 mm Hg at the end. The freeze drying time was approx. 10 hours, which resulted in less than 1.2% residual sample moisture. At this stage, the vacuum was broken with nitrogen, and the samples were taken out in air and packaged in laminated foil pouches under nitrogen, until ready for equilibration. Fig. 12 shows a number of the dehydrated meat samples which were used for the experimental work.

Equilibration of meat samples at different moisture content - water vapor equilibrium conditions.

The selection of equilibrium conditions for the meat samples was based on the moisture sorption isotherm, taking under consideration anticipated conditions of storage and utilization of these special foods. An effort was made to cover main points of the isotherm below, at, and above the B.E.T. monomolecular layer of water, without exceeding the lowest limit of growth for most microorganisms. The conditions selected were equilibration under reduced pressure in desiccators at 20°C , under 0, 12, 23 and 66% R.H., using $\text{Mg}(\text{ClO}_4)_2$, and saturated solutions of LiCl , CH_3COOK and NH_4NO_3 , respectively. The desiccators were evacuated to a pressure

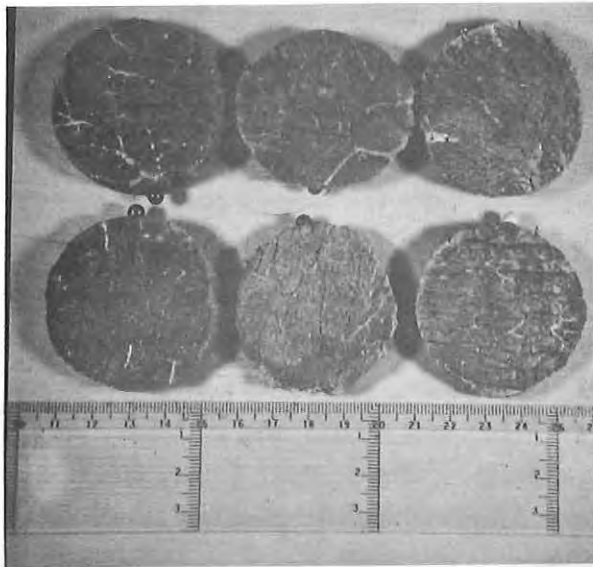


Fig. 12. Photo of samples of pre-cooked freeze-dried beef, S.I.K. series.

close to that of the corresponding constant R.H. medium.

The meat samples were distributed over the different humidities according to Tables 5 and 6. The purpose for this sample distribution was to: (a) discount, as much as possible, textural variability due to location of the meat within the muscle of each animal, (b) ensure testing by the Masticometer at different penetration depths, whereby the rheological behavior of meat through complete chewing cycles could be studied, and (c) ensure a sufficient number of replicate measurements, whereby the effect on texture of the relative humidity could be statistically analyzed.

Periodically upon storage, the loss or gain in moisture by the samples was determined by weighing. Equilibration was practically complete after about $2\frac{1}{2}$ months. However, the samples were not tested by the Masticometer until a total of $5\frac{1}{2}$ months under the above conditions. This time allowed for the first "setting" in texture, which is normally experienced in meat upon storage. All instrumental, sensory and other measurements performed on the samples reflect textural characteristics under these realistic conditions. (The rheological properties of meat as affected by moisture immediately upon processing are of more theoretical interest, and they may form the subject of useful supplementary studies to the present work in the future).

Rehydration Behavior

The meat was rehydrated in distilled water at 27°C for 20 minutes. The samples were placed on the surface of the water, and they were allowed to absorb water and gradually sink by their own weight. After 20 minutes, they were removed from the water, excess moisture was removed by blotting on brown paper, and the meat was weighed on a torsion balance. Results are expressed as

(a) % total moisture on Non-fat dry (N.F.D.) matter

$$\frac{\text{g. total moisture}}{\text{g. non-fat dry matter}} \times 100$$

Table 5. Systematic distribution over different relative humidities of samples from semimembranosus muscle, S.I.K. I and II.

Side L					R				
Circle depth	a		b		Circle depth	a		b	
	D ₁	D ₃	D ₂	D ₄		D ₁	D ₃	D ₂	D ₄
Slice					Slice				
1	*		+		1		o		x
2	+		*		2		x		o
3	o		x		3		*		+
4	x		o		4		+		*
5		*		+	5	o		x	
6		+		*	6	x		o	
7		o		x	7	*		+	
8		x		o	8	+		*	
9	*		+		9		o		x
10	+		*		10		x		o
11	o		x		11		*		+
12	x		o		12		+		*
13		*		+	13	o		x	
14		+		*	14	x		o	
15		o		x	15	*		+	
16		x		o	16	+		*	

L=left side of muscle, R=right side of muscle; a, b=pieces of same slice

* = 0% R.H. /Mg (ClO₄)₂/

+ =12% R.H. /LiCl/

o =23% R.H. /CH₃COOK/

x =66% R.H. /NH₄NO₃/

D₁ - D₄ = depth of penetration, each measurement in duplicate on the dry sample. Each sample was subsequently rehydrated and penetrated in duplicate at the corresponding D₁ etc. level.

Table 6. Systematic distribution over different relative humidities of samples from semimembranosus muscle, S.I.K. III and IV.

Side L							R						
Circle		a		b			a						
Depth	D ₁	D ₂	D ₁	D ₂			D ₁	D ₂		D ₁	D ₂		
Sensory evaluation		S		S			S			S			
Slice							Slice						
1	*				+		1		o		x		
2			+			*	2			x		o	
3	o				x		3		*		+		
4			x			o	4			+		*	
5		*		+			5	o			x		
6			+			*	6			x		o	
7		o		x			7	*			+		
8			x			o	8			+		*	
9	*				+		9		o		x		
10			+			*	10			x		o	
11	o				x		11		*		+		
12			x			o	12			+		*	
13		*		+			13	o			x		
14			+			*	14			x		o	
15		o		x			15	*			+		
16			x			o	16			+		*	

L = left side of muscle, R = right side of muscle, a, b = pieces of same slice

* = 0 % R.H. /Mg (ClO₄)₂/

+ = 12 % R.H. /LiCl/

o = 23 % R.H. /CH₃COOK/

x = 66 % R.H. /NH₄NO₃/

D₁, D₂ = depth of penetration, each measurement in duplicate on the dry sample.

Each sample was subsequently rehydrated and penetrated in duplicate at the corresponding D₁, D₂ level

S = Sensory evaluation of the dry sample

(b) coefficient of weight restoration (x 100)

$$\frac{r \text{ of meat after rehydration}}{S \text{ of meat before rehydration}} \times 100$$

III. RAW FREEZE-DRIED BEEF PROCESSED BY S.I.K.

Although the study of the textural quality of raw freeze-dried beef was not included in the proposal originally submitted for this Fellowship, certain experiments were considered desirable. Exact knowledge of comparative textural characteristics of raw freeze-dried meat in the dry and reconstituted states is missing, especially with regard to the methodology applied in this Report. Such knowledge may lead to further research on the objective measurement of the textural parameters of raw freeze-dried meat, a product of considerable military importance.

The meat was representative of semimembranosus muscle with 8 % fat on dry basis, from an animal 2 - 3 years old, of a grade corresponding to U.S. Good. It was processed by the Pilot plant of S.I.K., by first freezing in a freezer chamber at - 30°C for 17 hours, and then dehydrating under the same conditions as for the pre-cooked meat. The samples were packaged in pairs of rectangular or kidney-like slices, of approximate maximum dimensions 70x40x10 mm, in laminated foil pouches under nitrogen. This meat was tested by the Masticometer in the dry (1 % moisture on N.F.D. basis) and in the reconstituted state, with no intermediate humidity conditions studied. Each slice was cut in half, one piece serving for measurements on the dry and the other for measurements on the rehydrated material.

IV. SPECIAL DEHYDRATED FOODS SUPPLIED BY THE U.S. ARMY NATICK LABORATORIES (NLABS)

Research work on these foods was also not planned in the original proposal. The textural characteristics of such special foods as affected by the moisture content - water vapor equilibrium,

were largely unknown, and the other work on this Fellowship provided a good opportunity for an additional study. These foods are used mainly for space feeding, and detailed accounts on their processing, uses and other aspects have been published recently in the literature (46, 54, 83).

The foods were supplied by NLABS, packaged under nitrogen in cans of different sizes. They included the following items:

Beef sandwich	uncoated
Chicken sandwich	"
Cheese sandwich	"
Chicken bites	"
Melba toast	"

Of the above items, the melba toast was exceedingly hard, and impossible to chew by a sensory panel. This, and certain other foods not mentioned in the above list, were excluded from texture measurements. The special foods were equilibrated under the same relative humidity conditions which were described for the pre-cooked freeze-dried beef processed by S.I.K., i.e. 0, 12, 23 and 66 % R.H. at 20°C.

V. PRE-COOKED DEHYDRATED BEEF PROCESSED BY NLABS

This meat was processed by the Animal Products Branch of NLABS under three different dehydration procedures, viz. freeze-drying, air-drying, and freeze-desiccation (Dunston process). It included "U.S. Good" rib eye, and "U.S. Commercial" boneless top round. All meat was trimmed of fat and connective tissue, and it was cooked by steam at a pressure of 6 lbs/sq.in. Table 7 describes the types of meat used and the processing methods followed.

After cooking, the meat was cooled and sliced by an electric slicer to 1/4 inch thick pieces. Freeze-drying (lots 1, 2, 5 and 1A) was done by using 130°F final plate temperature, to about 1 % residual moisture. Air drying (lot 4) was done at 14 % R.H. and 135°F dry heat, for 20.5 hours, to 9.5 % residual moisture. Each

Table 7. Pre-cooked dehydrated meat processed by NLABS

NLABS	Sample code		Rib eye	Round	U.S. Good	U.S. Commercial	Cooking time min.			Freeze-dried	Freeze- desiccated (Dunston)	Air-dried
	This Report						80	115	135			
1	I	X			X		X			X		
2	II	X			X			X		X		
3	III	X			X		X				X	
4	IV	X			X		X					X
5	V			X		X			X	X		
1A	IA	muscle 3			X		X			X		
3A	IIIA	muscle 1			X		X				X	

rib eye muscle from lots 1 and 3 was separated into two half portions. One half of lot 1 (designated 3A) was dehydrated as lot 3 by freeze-desiccation (Dunston process), while the other half (designated 1) was freeze-dried. One half of lot 3 (designated 1A) was freeze-dried, while the other half (designated 3) was freeze-desiccated. The meat samples were packaged in four No 2 $\frac{1}{2}$ cans under nitrogen.

The above samples were processed by NLABS on request by the recipient, soon after the award of this Fellowship, in order that maximum utilization of time upon arrival in Sweden could be made. However, when the cans were opened at S.I.K., most slices were found fragmented into pieces, and the individuality of each sample as to the position in the muscle, uniformity of cooking, thickness and other factors was lost. All these aspects were important with regard to the statistical design and the information sought for in the present study. It was therefore decided to process a new series of samples by the Pilot plant of S.I.K. As described earlier in this Report, the S.I.K. processed samples constitute the main experimental material of this work. However, the samples processed by NLABS were also equilibrated and fully tested by the Masticometer under conditions similar to those applied for the S.I.K. sample series. The Masticometer parameters of the NLABS samples were calculated on the basis of more than 500 different curves, and the data were filed for future analysis and interpretation. It is possible that meaningful results can be derived from these data, if properly analyzed and interpreted.

VI. DETERMINATION OF MOISTURE SORPTION ISOTHERMS

Moisture sorption isotherms were determined for all foods at 4°, 10°, 15°, 20°, and 30°C, using the weight equilibrium method. This method was best suited to the time schedule of this Report, and to the necessity of using larger, representative samples of the highly heterogeneous materials (sandwiches, etc.) of this study. For this purpose, about 5 g quantities of ground

food in weighing bottles were equilibrated inside desiccators at the desired temperature, using H_2SO_4 solutions of different specific gravity, $\text{Mg}(\text{ClO}_4)_2$ and saturated salt solutions, as shown in Table 8. The values of % relative humidity corresponding to the R.H. controlling media were taken from the literature (47, 74, 87, 112), except for the saturated NH_4NO_3 solution, whose % R.H. at the different temperatures was determined by NLABS. A mechanical pump was used to evacuate the air in the desiccators and reduce the vapor pressure to a level close to that provided by the corresponding constant relative humidity medium. Periodically, the samples were weighed on an analytical balance until equilibrium was reached, which usually necessitated between three and five weeks. After equilibrium, the moisture and fat content of each sample were determined by the Analytical Laboratory of S.I.K., using the 17-hours vacuum oven method for moisture, and the 6-hour diethyl ether Soxhlet extraction method for fat. (Analytical measurements after equilibrium independently for each sample were decided, since a 100°C standard oven moisture method, applied before equilibrium, gave too high results for the calculation of the final moisture contents by difference in weight). The specific gravities of the H_2SO_4 solutions were also determined after equilibrium, and the relative humidities corresponding to these values were used for the plotting of the moisture sorption isotherms.

VII. CALCULATION OF PHYSICO-CHEMICAL VARIABLES

On the basis of the moisture sorption isotherms, determined at the temperatures mentioned above, the following work was performed:

- 1) Calculation of the B.E.T. value for a monomolecular layer of water, according to the B.E.T. equation (1, 7, 8)

Table 8. Values of % relative humidity used for the determination of moisture sorption isotherms

R.H. controlling agent	Temperature				
	4°C	10°C	15°C	20°C	30°C
$\text{Mg}(\text{ClO}_4)_2$	0	0	0	0	0
H_2SO_4					
d=1.608 g/ml	3.8				
d=1.618 g/ml		3.7			
d=1.606 g/ml			4.0		
d=1.582 g/ml				6.4	
d=1.680 g/ml					2.2
LiCl	16	14	13	12	11
CH_3COOK	24	24	24	23	23
MgCl_2	33	33	33	32	32
K_2CO_3	50	47	45	44	42
NH_4NO_3	74	72	70	66	60
NaCl	75	75	75	75	75

$$\frac{p}{a (p_0 - p)} = \frac{1}{a_1 \cdot c} + \frac{c - 1}{a_1 \cdot c} \cdot \frac{p}{p_0}$$

(equation 1)

where:

a = g of water per 100g dry solids at moisture vapor pressure p ,

p_0 = vapor pressure of pure water at the same temperature,

c = constant related to the heat of adsorption,

a_1 = g of water equivalent to the monomolecular layer adsorbed on 100 g. of dry solids.

In this Report the following convenient form of the B.E.T. equation was used (88)

$$\frac{R}{a (100 - R)} = I + S \cdot R \quad (\text{equation 2})$$

where:

R = percent relative humidity

a = defined as above

I = y-axis intercept, and S = slope of the straight line plot of $\frac{R}{a (100 - R)}$ against R .

The value of a_1 of the monomolecular layer was calculated by the equation:

$$a_1 = \frac{1}{I + 100S} \quad (\text{equation 3})$$

On the basis of the B.E.T. monomolecular layer of water, the surface area was calculated (4, 5, 61, 76, 92), using the value of 10.8 \AA^2 as the cross-sectional area of the water molecule.

2) Calculation of the Fugassi constants A , K , and K_1 , on the basis of the Fugassi isotherm equation (32-34, 58, 73, 77),

$$W_e = \frac{AKK_1P_0C}{1 \left[(K_1P_0 - 1) C \right] (1 - C) + KK_1P_0C}$$

(equation 4)

where:

We = weight of water sorbed at relative pressure C,

A = total number of sorption sites, one sorbate molecule per site,

K = equilibrium constant for the transfer reaction from the surface to the interior of the sample,

K₁ = equilibrium constant for the sorption from the vapor phase onto the surface,P₀ = saturation vapor pressure,C = relative vapor pressure = P/P₀

This equation has three independent variables for a given system, A, K, and K₁, which are defined in the following way:

$$\frac{C}{W_e} = a + bC - \gamma C^2 \quad (\text{equation 5})$$

where:

$$a = \frac{1}{AKK_1P_0} \quad (\text{equation 6})$$

$$b = \frac{KK_1P_0 + K_1P_0^{-2}}{AKK_1P_0} \quad (\text{equation 7})$$

and

$$\gamma = \frac{K_1P_0^{-1}}{AKK_1P_0} \quad (\text{equation 8})$$

Simultaneous solution of a set of three equations like eq. 5 leads to values of a, b and γ. The three isotherm parameters are then obtainable through the relationships

$$\Lambda = \frac{1}{a + b - \gamma} \quad (\text{equation 9})$$

$$K = \frac{a + b - \gamma}{a + \gamma} \quad (\text{equation 10})$$

and

$$K_1 = \left(\frac{\gamma}{a} + 1 \right) / P_0 \quad (\text{equation 11})$$

The above Fugassi constants were calculated by a high speed computer at the Data Analysis Branch of the U.S. Army Natick Laboratories. The computer program was kindly supplied by Dr. N. Laine of the Melpar Company, Falls Church, Virginia (58).

3) Calculation of the partial molal heat of sorption $\overline{\Delta H}$ according to the Clausius - Clapeyron equation

$$\ln p = \frac{-\overline{\Delta H}}{R} \cdot \frac{1}{T} + C \quad (\text{equation 12})$$

where p is the partial vapor pressure of water at temperature T within the range of interest, and R the gas constant. In practice, isosteric values of p on a log scale were plotted against $\frac{1}{T}$ on a linear scale, using the data from the isotherms at different temperatures. The slope of any isostere is equal to $-\overline{\Delta H} / 2.3 R$. The $\overline{\Delta H}$ obtained from this plot is the sum of $\overline{\Delta H}_{\text{adsorption}} + \overline{\Delta H}_{\text{condensation}}$ of water. The $\overline{\Delta H}_{\text{net}}$ was obtained by subtracting the $\overline{\Delta H}_{\text{condensation}}$ from the $\overline{\Delta H}$ value of the above plot.

The net integral heat change was calculated (18, 22) using the equation:

$$\Delta H = \frac{RT_1 T_2}{T_2 - T_1} \int_0^n \frac{x_1}{x_2} \cdot dn \quad (\text{equation 13})$$

where x_2 is the relative vapor pressure at the higher temperature T_2 which produces the same number of moles of water sorption as at

the lower temperature T_1 for which x_1 applies.

4. Calculation (9) of the partial molal free energy change as follows:

$$\bar{\Delta} G = \frac{dG}{dn} = RT \ln x \quad (\text{equation 14})$$

The integral free energy change for water vapor accompanying the sorption process was calculated (18, 22) using the equation:

$$\Delta G = - RT \int_0^1 n d \ln (p/p_0) \quad (\text{equation 15})$$

where n is the number of moles of water sorbed per 100 g of non-fat dry matter. In this process p/p_0 is considered the independent variable.

The integration of the right-hand side of the above equation was carried out as follows:

$$\int_0^1 n d \ln (p/p_0) = \int_0^1 n p_0/p \cdot d (p/p_0) \quad (\text{equation 16})$$

A plot of $n p_0/p$ vs p/p_0 was made, and the total area under the curve multiplied by RT gave the integral free energy of sorption.

5) Calculation of the corresponding entropy changes $\bar{\Delta} S$ and ΔS as follows:

$$\bar{\Delta} S = (\bar{\Delta} H - \bar{\Delta} G) / T \quad (\text{equation 17})$$

$$\Delta S = (\Delta H - \Delta G) / T \quad (\text{equation 18})$$

SENSORY EVALUATION

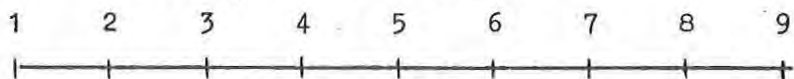
The sensory evaluation work was conducted by the staff of the Experimental Kitchens of S.I.K., and it applied to the pre-cooked freeze-dried beef S.I.K. III and IV sample series.

Training of the sensory panel and design of experiments on sensory evaluation

Eight subjects were asked to participate in the sensory evaluation. Of these, only two had some previous experience in tasting freeze-dried meat in the dry state. In order to get acquainted with this new product, the training was started by merely chewing and tasting the dry samples, without rating any properties of the material. After a number of sessions, the panel decided that it would be meaningful to judge the hardness of the meat by biting both parallel to the fibers and across them. The procedure finally selected was to evaluate the hardness by (a) biting with the molars over a piece of dry meat as it was (=parallel to the fibers), and (b) biting with the incisors across a bundle of fibers taken from the sample. This bundle of fibers was about 3 mm thick. The samples were rated by using a 9-point unstructured scale with the end points "extremely difficult to bite through" to "extremely easy to bite through", (cf., sheet on next page).

After the meat had been rehydrated in the mouth, the total impression of flavor and tenderness was rated by using a 9-point scale of the same kind as the one used for hardness.

For each one of the above mentioned properties "hardness parallel to fibers", "hardness across fibers", "flavor" and "tenderness", the scale divisions were given numbers from 1 (left end) to 9 (right end) as follows:



Mean values of scores were then calculated for each combination of humidity, judge and taste session. Differences between these mean values were significance-tested by applying Analysis of Variance.

Example of instruction sheet with rating scales for the sensory evaluation of pre-cooked freeze-dried beef, S.I.K. III and IV.

Type: _____ For _____ Date _____ 19 _____

Judge _____ Session: _____ Sample _____

In this session you will receive _____ samples, of which this is No. _____. The samples will be served one at a time. The taste testing will be carried out according to the attached instructions and the scales below.

HARDNESS BEFORE REHYDRATION IN THE MOUTH

a) When you bite with the molars paralalled to the fibers

extremely difficult
to bite through

extremely easy
to bite through



b) When you bite with the incisors across the fibers

extremely difficult
to bite through

extremely easy
to bite through



HEAT EVOLUTION DURING REHYDRATION IN THE MOUTH

None

Insignificant

Evident

Very evident

PROPERTIES AFTER REHYDRATION IN THE MOUTH

a) Flavor

extremely
bad

extremely
good



b) Tenderness

extremely
tough

extremely
tender



Remarks: _____

Sign: _____

At the training sessions, the sensory panel noticed that the dry meat caused a certain evolution of heat in the mouth when it was allowed to rehydrate with the saliva. It was, therefore, decided to rate the amount of heat evolved during mastication, by using a scale from "no heat evolution" to "very evident heat evolution", as follows:

0 = none, 1 = insignificant 2 = evident 3 = very evident

For each relative humidity, arithmetic means of the given scores were calculated and plotted as a function of different variables.

STATISTICAL DESIGN OF INSTRUMENT DATA

S.I.K. I and II sample series

For each variable investigated, Analysis of Variance was performed on sets of data according to the following design (cf., Tables 5 and 6)

Humidity	Position and depth							
	P ₁ (left)				P ₂ (right)			
	D ₁	D ₂	D ₃	D ₄	D ₁	D ₂	D ₃	D ₄
H ₁	r ₁	r ₁	r ₁	r ₁	same			
	r ₂	r ₂	r ₂	r ₂				
	r ₃	r ₃	r ₃	r ₃				
	r ₄	r ₄	r ₄	r ₄				
H ₂	same				same			
H ₃	same				same			
H ₄	same				same			

r = replicate, other letters as in Tables 5 and 6

In the statistical analysis, the variance for all factors (H, P and D) and their interactions (HP, HD, PD, HPD) were calculated and tested against the error variance (=mean square for "within replicates").

S.I.K. III and IV sample series

The same analysis was applied as above but the number of depths was 2 instead of 4.

The Analyses of Variance were all done on the basis of a computer program by the I.B.M. Data Center, Stockholm.

STATISTICAL DESIGN OF SENSORY DATA

The distribution of samples used in the sensory evaluation was given in Table 6, which also explained the meaning of the four code signs used. As shown in this Table, 4 pieces of meat were available for each humidity from each one of the left and right muscle. Each piece was cut into two parts, which provided twice as many samples for sensory evaluation.

Four persons, who had successfully participated in all the training sessions, were chosen as a sensory panel. The order in which the samples were served in each taste session was as follows:

S.I.K. III sample series

Taste session	Order of serving of the samples for:															
	Judge 1				Judge 2				Judge 3				Judge 4			
	L		R		L		R		L		R		L		R	
I	*	+	o	x	+	*	x	o	o	x	*	+	x	o	+	*
II	+	*	x	o	o	x	*	+	x	o	+	*	*	+	o	x
III	o	x	*	+	x	o	+	*	*	+	o	x	+	*	x	o
IV	x	o	+	*	*	+	o	x	+	*	x	o	o	x	*	+

S.I.K. IV Sample series

Taste session	Order of serving of the samples for:															
	Judge 1				Judge 2				Judge 3				Judge 4			
	L		R		L		R		L		R		L		R	
I	*	+	o	x	+	x	*	o	o	*	x	+	x	o	+	*
II	+	x	*	o	o	*	x	+	x	o	+	*	*	+	o	x
III	o	*	x	+	x	o	+	*	*	+	o	x	+	x	*	o
IV	x	o	+	*	*	+	o	x	+	x	*	o	o	*	x	+

For each one of the S.I.K. III and IV sample series, slices 2 and 4 from both the left and right muscles (cf., Table 6), were used for taste session I; slices 6 and 8 for taste session II; 10 and 12 for taste session III; and slices 14 and 16 for taste session IV.

RESULTS AND DISCUSSION

MOISTURE SORPTION ISOTHERMS

Equilibrium values determined for the moisture sorption isotherms at the different temperatures are shown in Tables 9 - 17. The short temperature range, 4-30°C, which was of interest in this work resulted in closely spaced isotherms. Plots of the data of 4 and 30°C are presented in Figs. 13-21. The isotherms were generally of the sigmoid type, and their relative position at the different temperatures agreed with the work reported recently by Saravakos and Stinchfield (91). The data indicated smaller differences among samples I, II, III, and IV, in comparison with the special foods. In most of the latter foods, it is interesting to note the cross-over of the isotherms at a relative humidity which is close to that corresponding to the completion of a second layer of water according to the B.E.T. theory. This is probably due to the influence of the non-protein components of the mixture.

Table 9. Equilibrium values for moisture sorption isotherms of pre-cooked freeze-dried beef, S.I.K. I

Relative vapor pressure p/p_0	Moisture g/100g N.F.D. ^u				
	4°C	10°C	15°C	20°C	30°C
0.05	2.23	2.23	1.69	1.52	1.28
0.10	3.04	3.10	2.47	2.39	1.74
0.20	4.16	3.95	3.44	3.44	2.78
0.30	5.09	4.69	4.40	4.39	3.89
0.40	6.02	5.63	5.51	5.49	5.06
0.50	6.90	7.09	7.09	6.98	6.60
0.60	8.68	9.30	9.11	8.92	8.87
0.70	12.53	12.68	12.25	12.00	12.72
0.75	16.40	14.82	14.40	14.01	14.48

Table 10. Equilibrium values for moisture sorption isotherms of pre-cooked freeze-dried beef, S.I.K. II

Relative vapor pressure p/p_0	Moisture g/100g N.F.D.				
	4°C	10°C	15°C	20°C	30°C
0.05	2.04	1.93	1.76	1.69	1.19
0.10	3.01	2.98	2.50	2.45	1.75
0.20	4.41	4.04	3.43	3.28	2.82
0.30	5.42	4.67	4.39	4.19	3.80
0.40	6.16	5.48	5.50	5.32	4.95
0.50	6.87	6.80	6.90	6.89	6.43
0.60	8.60	8.79	9.12	9.06	8.89
0.70	12.73	12.60	12.75	12.52	12.76
0.75	17.23	15.07	15.30	14.95	15.52

Table 11. Equilibrium values for moisture sorption isotherms of pre-cooked freeze-dried beef, S.I.K. III

Relative vapor pressure p/p_0	Moisture $g/100g_{N.F.D.}$				
	4°C	10°C	15°C	20°C	30°C
0.05	2.30	2.22	1.62	1.69	1.12
0.10	3.11	3.10	2.34	2.50	1.63
0.20	4.26	4.18	3.22	3.30	2.67
0.30	5.16	4.97	4.19	4.09	3.72
0.40	5.83	5.72	5.40	5.39	4.86
0.50	6.80	6.79	6.66	7.02	6.34
0.60	8.60	8.60	8.32	9.29	8.69
0.70	12.70	12.50	12.18	12.70	12.50
0.75	17.50	15.32	15.35	15.05	15.72

Table 12. Equilibrium values for moisture sorption isotherms of pre-cooked freeze-dried beef, S.I.K. IV

Relative vapor pressure p/p_0	Moisture g/100g N.F.D.				
	4°C	10°C	15°C	20°C	30°C
0.05	2.00	2.26	1.80	1.60	1.11
0.10	2.84	3.06	2.67	2.30	1.65
0.20	4.12	4.10	3.70	3.19	2.69
0.30	5.10	4.93	4.55	4.09	3.79
0.40	5.89	5.78	5.57	5.30	4.94
0.50	6.78	6.78	6.83	7.10	6.40
0.60	8.24	8.35	8.82	9.22	8.71
0.70	12.35	11.98	12.13	12.74	12.68
0.75	17.60	14.81	15.30	15.07	15.78

Table 13. Equilibrium values for moisture sorption isotherms of dehydrated beef sandwich

Relative vapor pressure p/p_0	Moisture g/100g N. F. D.				
	4°C	10°C	15°C	20°C	30°C
0.05	2.77	1.62	1.71	1.42	1.65
0.10	3.35	2.44	2.50	2.23	2.11
0.20	4.18	3.56	3.48	3.24	2.96
0.30	5.19	4.43	4.42	4.18	4.00
0.40	6.30	5.70	5.76	5.50	5.52
0.50	7.75	7.77	8.02	7.76	7.52
0.60	10.46	11.23	11.20	10.97	10.45
0.70	15.07	16.27	16.63	16.39	15.73
0.75	23.88	19.49	20.62	20.42	20.22

Table 14. Equilibrium values for moisture sorption isotherms of dehydrated chicken sandwich

Relative vapor pressure p/p_0	Moisture g/100g N. F. D.				
	4°C	10°C	15°C	20°C	30°C
0.05	2.78	2.78	2.49	2.27	1.78
0.10	3.54	3.73	3.40	3.25	2.36
0.20	4.67	4.62	4.27	4.15	3.22
0.30	5.79	5.38	4.92	4.71	4.29
0.40	6.73	6.61	6.55	5.80	5.61
0.50	8.02	8.37	8.10	7.80	7.62
0.60	10.26	11.33	11.96	10.59	10.75
0.70	15.52	15.73	15.28	15.09	16.24
0.75	24.20	18.62	17.86	18.38	21.10

Table 15. Equilibrium values for moisture sorption isotherms of dehydrated cheese sandwich

Relative vapor pressure p/p_0	Moisture g/100g N.F.D.				
	4°C	10°C	15°C	20°C	30°C
0.05	2.25	2.22	1.78	1.63	1.32
0.10	3.26	2.94	2.45	2.50	2.06
0.20	4.49	3.63	3.19	3.20	2.80
0.30	5.50	4.20	3.98	3.82	3.60
0.40	6.68	5.13	5.42	5.29	4.82
0.50	8.40	7.15	7.55	7.72	6.93
0.60	11.23	10.78	11.25	11.79	10.65
0.70	17.39	17.11	17.52	18.02	16.66
0.75	28.12	22.58	23.96	23.23	22.63

Table 16. Equilibrium values for moisture sorption isotherms of dehydrated chicken bites

Relative vapor pressure p/p_0	Moisture g/100g N.F.D.				
	4°C	10°C	15°C	20°C	30°C
0.05	3.40	2.50	2.72	2.42	2.00
0.10	4.18	3.53	3.55	3.34	2.65
0.20	5.28	4.88	4.41	4.21	3.46
0.30	6.18	5.72	5.22	5.00	4.11
0.40	7.23	6.78	6.57	6.49	5.65
0.50	8.47	9.45	9.52	9.50	9.45
0.60	11.05	14.25	14.53	14.73	15.05
0.70	19.82	22.56	22.45	23.05	22.74
0.75	34.51	30.41	30.50	29.49	28.01

Table 17. Equilibrium values for moisture sorption isotherms of
dehydrated melba toast

Relative vapor pressure p/p_0	Moisture g/100g N.F.D.				
	4°C	10°C	15°C	20°C	30°C
0.05	1.78	1.70	1.62	1.51	1.32
0.10	2.36	2.30	2.23	2.01	1.58
0.20	3.09	2.69	2.76	2.39	1.97
0.30	3.48	3.14	3.17	2.91	2.69
0.40	3.98	3.93	4.08	3.99	3.98
0.50	4.98	5.54	5.80	6.01	6.01
0.60	7.40	8.70	8.43	9.07	9.05
0.70	12.08	13.37	13.20	14.31	13.79
0.75	20.90	16.92	18.50	18.67	17.45

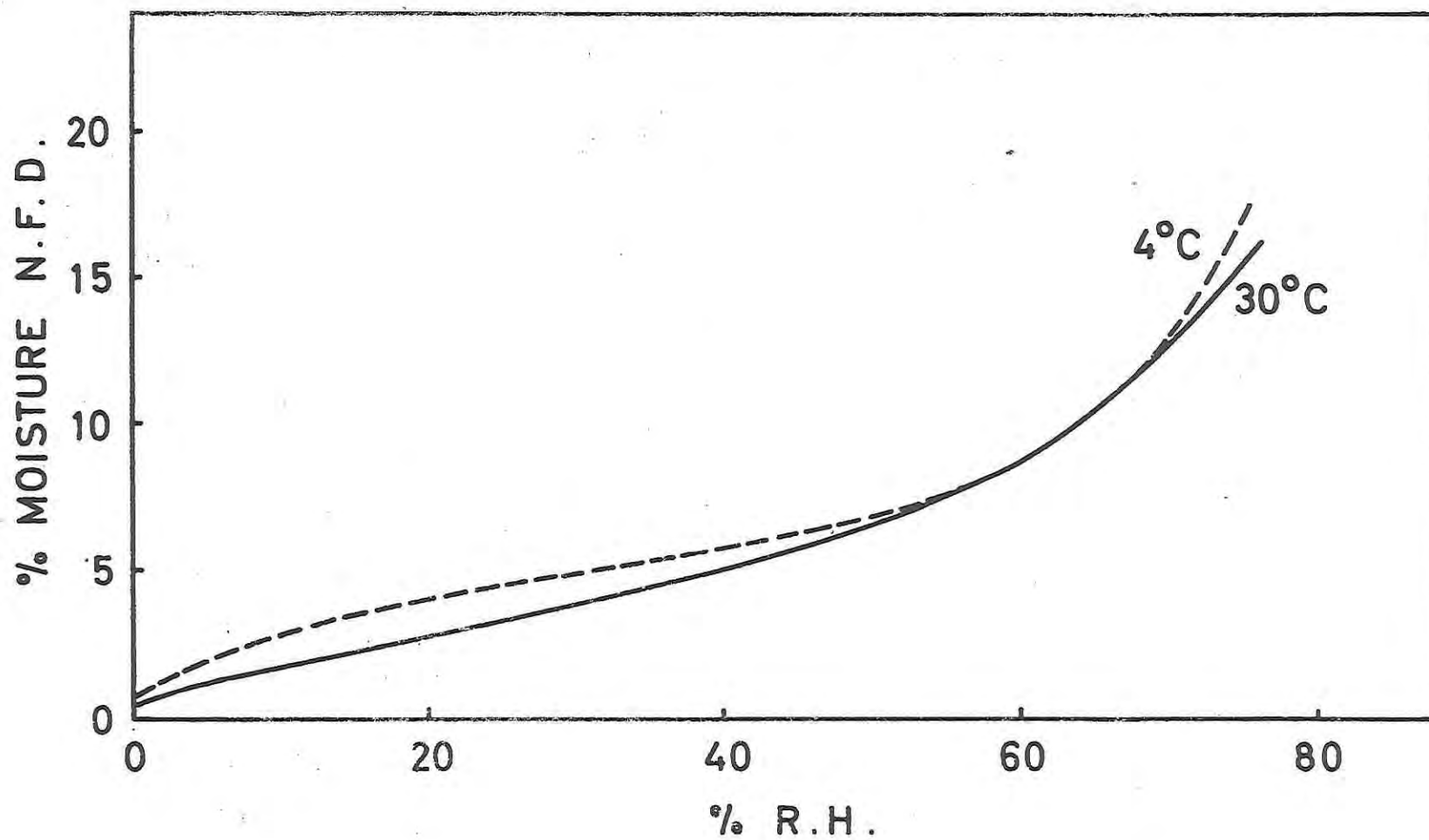


Fig. 13. Moisture sorption isotherms of pre-cooked freeze-dried beef, S.I.K. I.

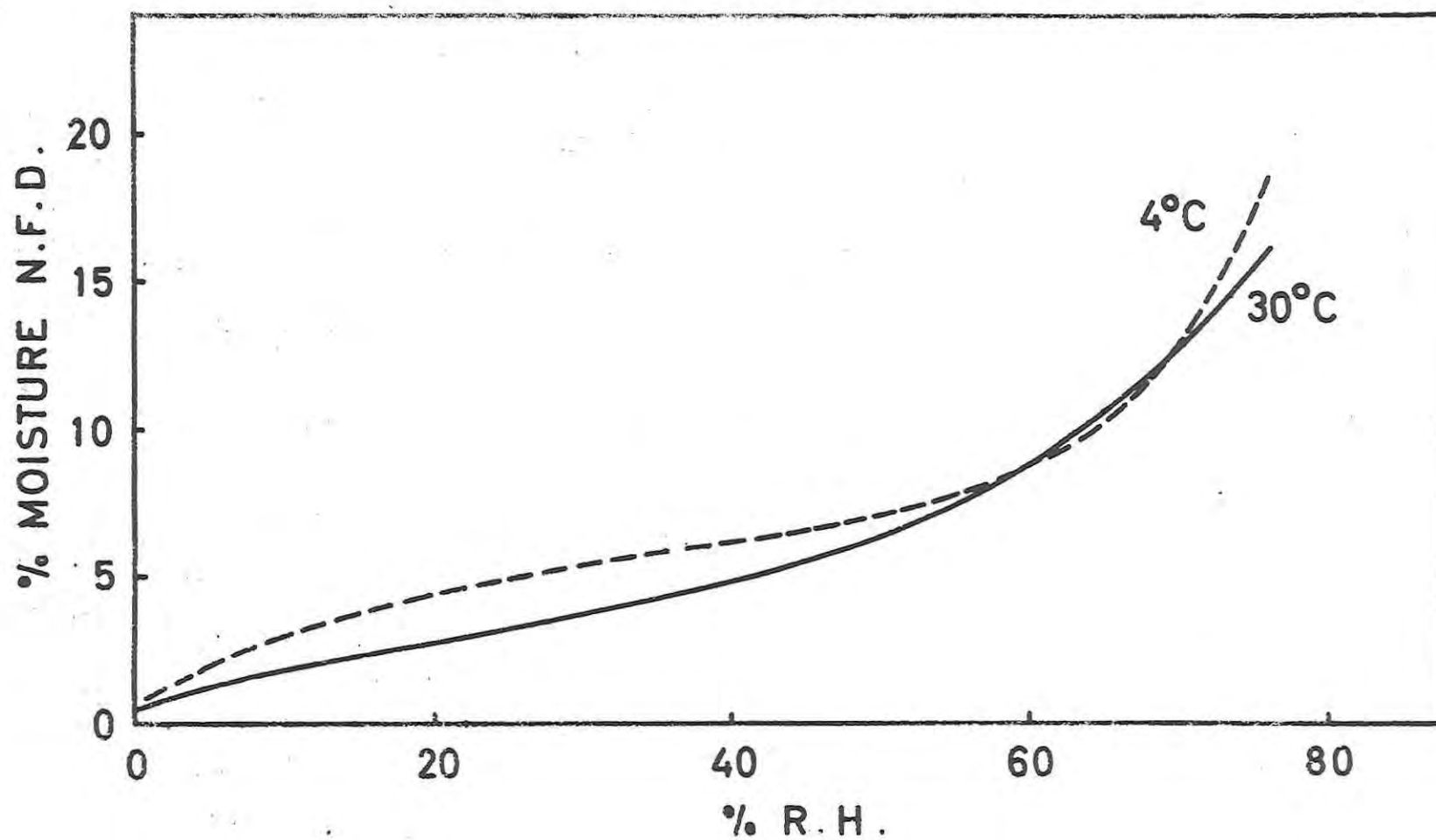


Fig. 14. Moisture sorption isotherms of pre-cooked freeze-dried beef. S.I.K. II.

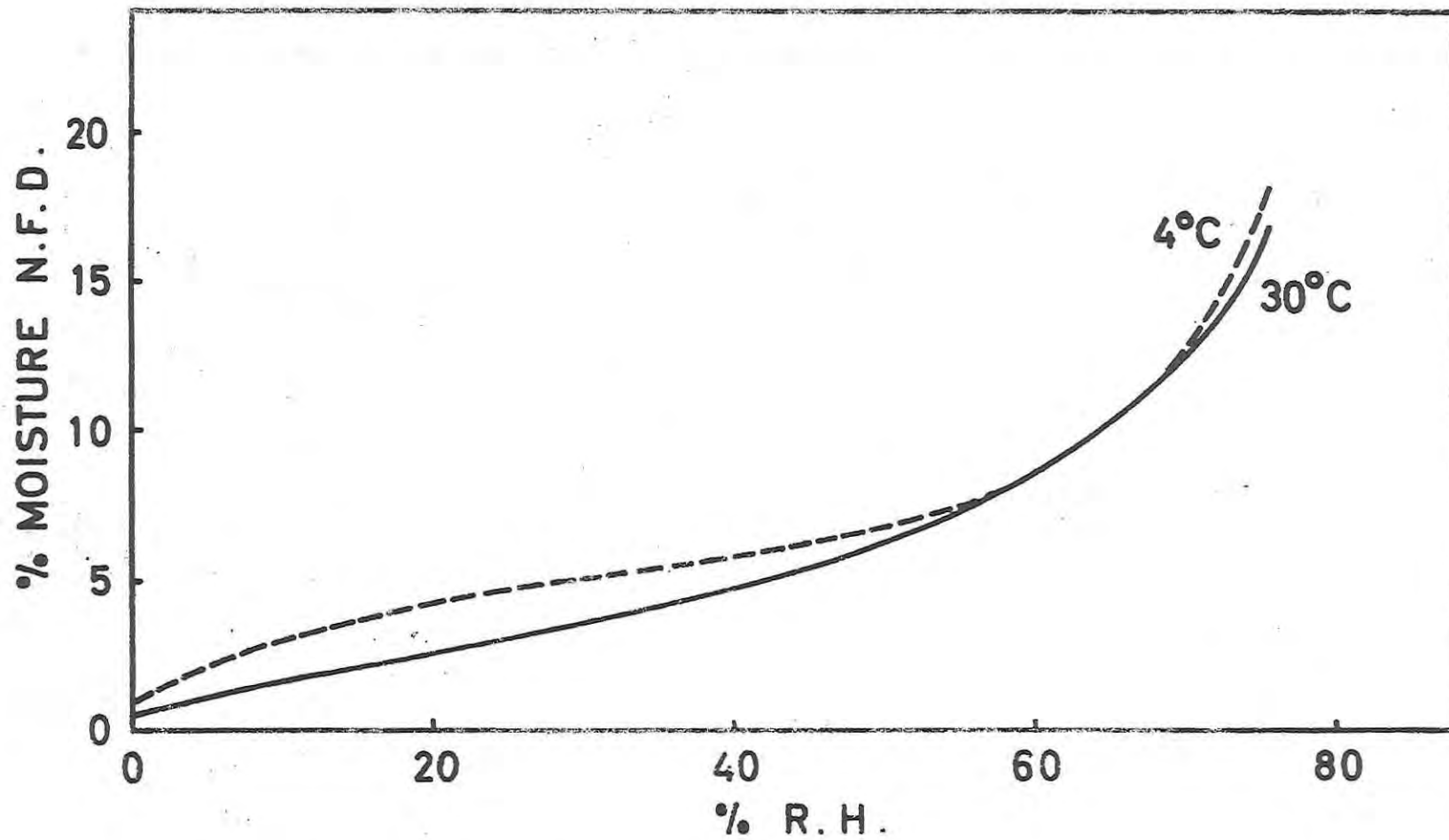


Fig. 15. Moisture sorption isotherms of pre-cooked freeze-dried beef, S.I.K. III.

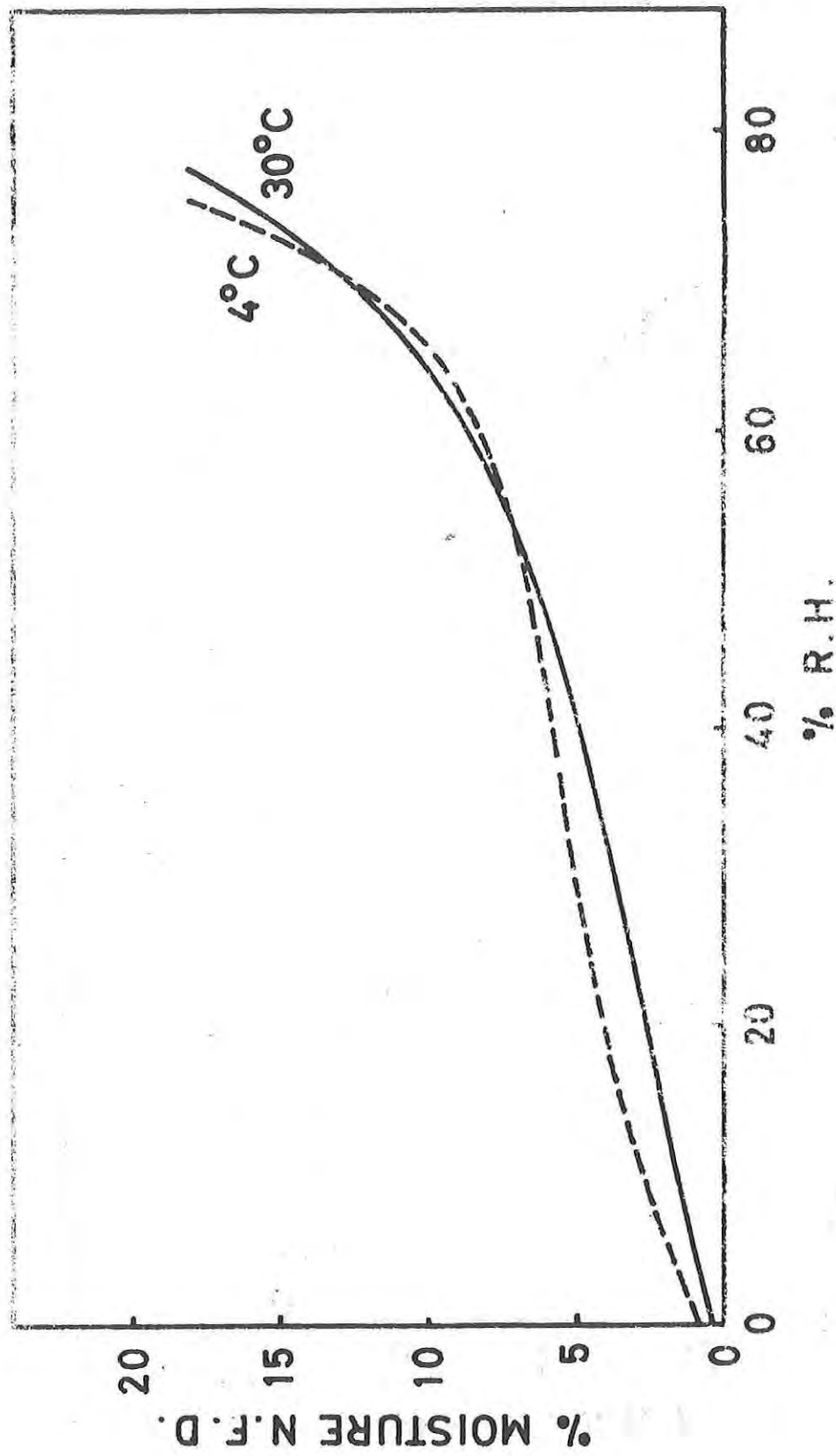


Fig. 16. Moisture sorption isotherms of pre-cooked freeze-dried beef, S.I.K. IV.

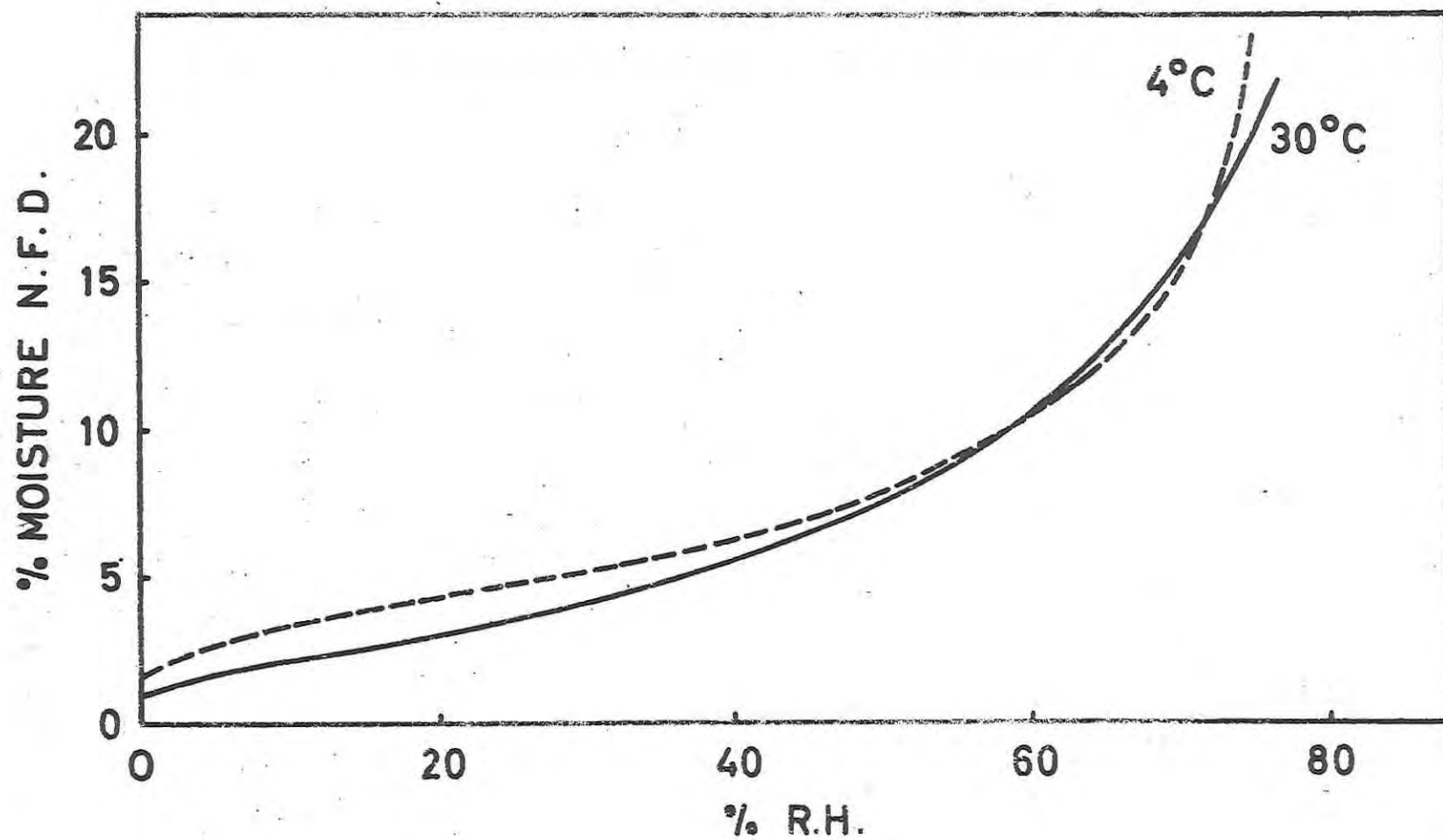


Fig. 17. Moisture sorption isotherms of dehydrated beef sandwich.

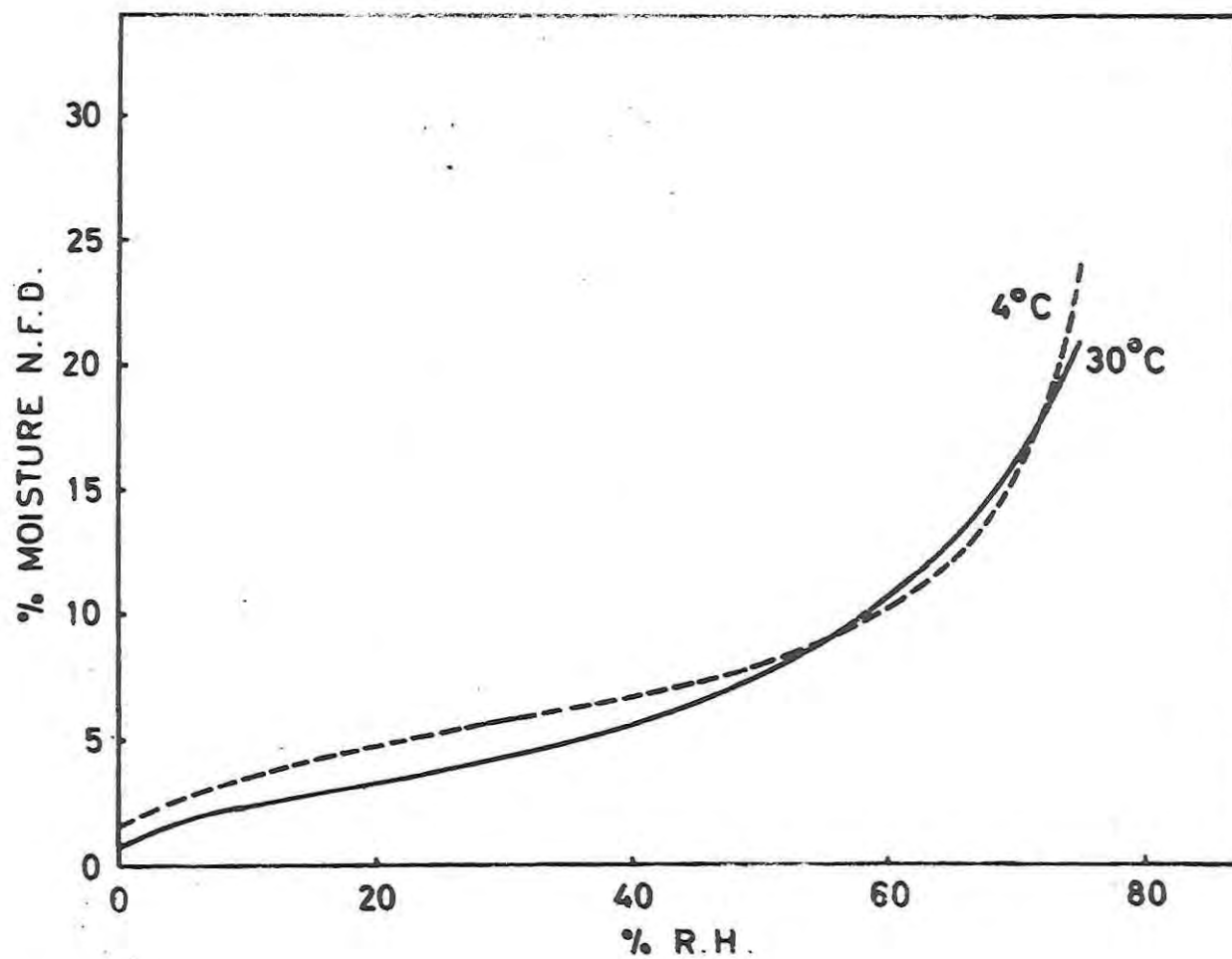


Fig.18.Moisture sorption isotherms of dehydrated chicken sandwich.

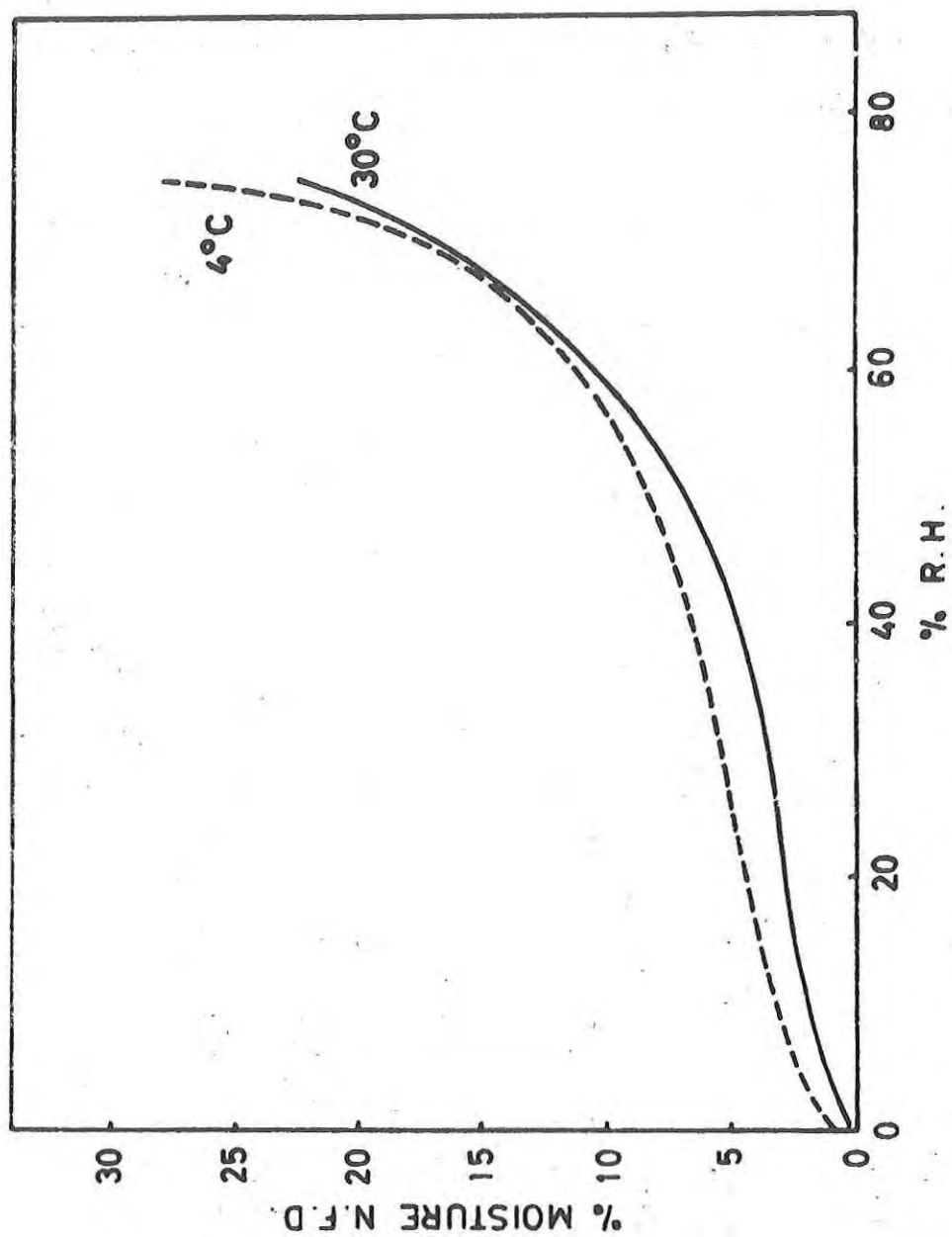


Fig. 19. Moisture sorption isotherms of dehydrated cheese sandwich.

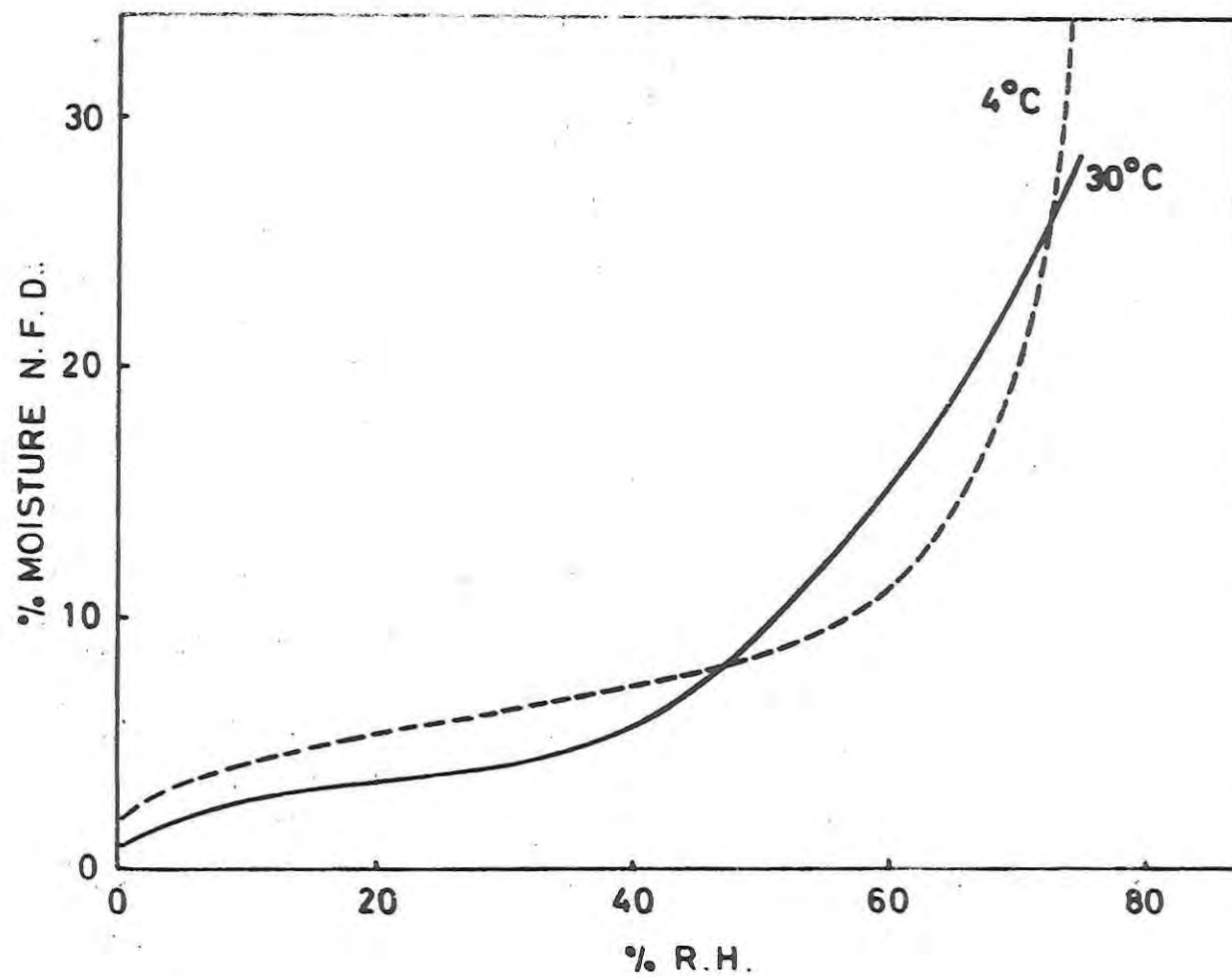


Fig. 20. Moisture sorption isotherms of dehydrated chicken bites.

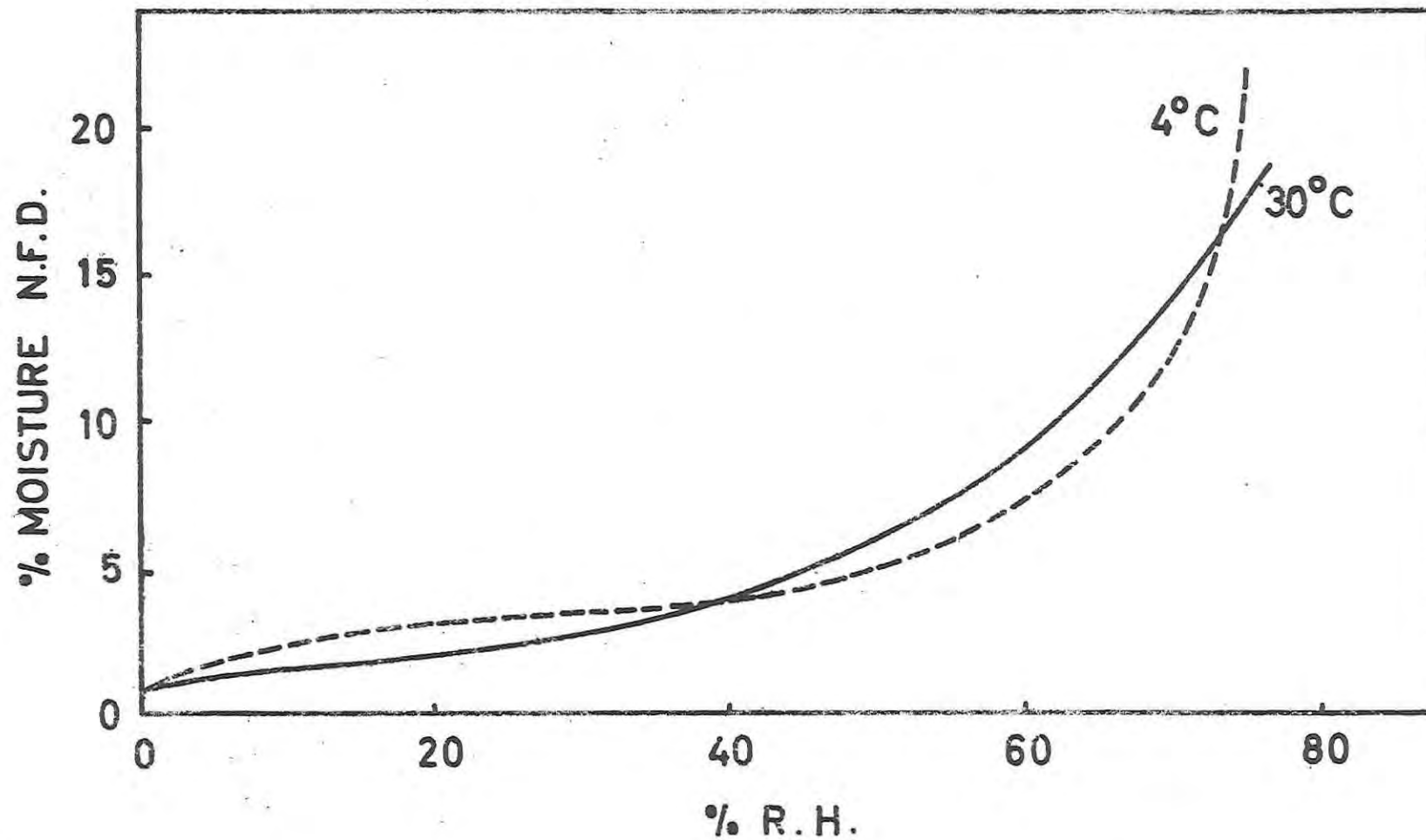


Fig. 21. Moisture sorption isotherms of dehydrated melba toast.

PRE-COOKED FREEZE-DRIED BEEF, S.I.K. 1-4 SAMPLE SERIES

Due to the large volume of data, the complete results on all S.I.K. I, II, III and IV series will be treated in the statistical Analysis of Variance. In this and other sections, the Tables, Figures and discussion will be limited to the S.I.K. II series which, in most cases, were representative of the information and trends reflected by the other data.

The relationship between penetration depth PD and relative instrument force KR for the dry samples, which had been stored at 20°C but at different relative humidities for 5½ months, is shown in Table 18 and Fig. 22. For clarity, only one "average" curve is shown. The individual curves which connect points of the same R.H. had similar characteristics, and extrapolated to zero, they generally had a sigmoid shape. From about zero to 40, and from 65 to 75% PD, there was a sharp increase of force as PD increased. Within these ranges, the selection of the depth of penetration for the objective measurement of hardness is important if comparable data are to be obtained. Between about 40 and 65% PD, the force changed less rapidly with PD, and the selection of PD is not so critical. The sharp increase of force above about 70% PD is probably due to the pressing or compacting of the meat under the punch. In special foods which are to be consumed without rehydration, this compactness associated with dryness, release of heat, crumbliness and other sensations during mastication pose new problems of texture to be investigated.

The relationship between penetration depth and relative instrument force for the same samples in the rehydrated state is shown in Table 19 and Fig. 23. In general, the instrument force increased continuously with PD, and instead of a sigmoid, a smooth, convex to both axes curve was obtained. As expected, the hardness of the rehydrated food was appreciably smaller than the hardness of the dry material.

The effect of the relative humidity on hardness is shown more clearly when the above data are plotted as in Fig. 24 for the dry, and in Fig. 25 for the rehydrated food. In the dry food, the increase of R. H. resulted in an appreciable increase of force. It is interesting to note that most of this increase took place above the B. E. T. monomolecular layer of water. This brings into focus the close relationship between chemical and textural changes. Above the B.E.T. monolayer, the less firmly bound water is more readily available for cross linking and other reactions which toughen the meat. At temperatures higher than 20°C and for periods longer than the 5½ months which were used in these experiments, this toughening should be even more pronounced. The above interpretation is supported by the work of Connell (10; 11) on the stress-strain behavior of cod muscle fiber bundles, and on the swelling behavior of finely divided muscle. From these and other experiments, the author concluded that toughness in freeze-dried rehydrated meats is associated with an increase of the number of bonds between actin and myosin. (Some denaturation of the actomyosin and myosin molecules, which occur to a greater extent in freeze dried fish than in beef, appears also to affect texture). During freeze-drying and storage, removal of the water which is normally interposed between the actin and myosin arrays, makes possible an increase in cross-linking between the two proteins. Hamm and Deatherage (40, 41), on the basis of experiments on the hydration and charge of freeze-dried rehydrated beef, and Webb (108) in his recent discussion of the relationships between bound water and molecular structure in biological systems, suggested analogous changes (see also ref. 60). The exact nature of the bonds formed is not presently clear. It is possible that hydrophobic bonding through mutual attraction of aliphatic sides of amino groups, as well as disulfide, amino-carbonyl and hydrogen bondings are involved. The increase of hardness brought about by the increase of relative humidity occurred also in the

rehydrated material, as shown in Fig. 25. The effect however, was much less pronounced in this case, especially at the lower penetration depths.

As stated previously, the force of penetration was much smaller in the rehydrated than in the dry food (Table 18, 19). Since in all cases of the dry material, the force increased with increasing relative humidity up to 66% R.H. used, there must be a turning point, beyond which the force decreases with relative humidity. This point probably occurs at much higher relative humidities, within the area of microbial deterioration. This fact is important in research and development efforts on foods which can be stable at elevated moisture levels. The Masticometer provides a quantitative means for the study of such changes, and it may serve as a valuable tool in efforts to improve the textural characteristics of dehydrated foods.

The elastic, crushing and total texture behavior of pre-cooked freeze-dried beef in the dry state, after equilibration at different relative humidities, is shown in Table 20, and Figs. 26-28. The same textural parameters of the meat in the rehydrated state are shown in Table 21 and Figs. 29-31. In general, the curves were sigmoid for the dry, and convex to both axes for the rehydrated material. In both the dry and the rehydrated samples, there was a tendency for an increase of the individual parameters as the relative humidity increased. The corresponding parameters were appreciably smaller for the rehydrated than for the dry material. In either case, the values for the elastic behavior were smaller than those for the crushing behavior, and this difference was appreciably greater in the dry than in the rehydrated food. This shows that most of the work of mastication is expended for irreversible crushing, especially in the case of the dry material. It is possible that in many special foods, substantial improvement of the textural quality can be effected by a modification of this parameter in its relationship to the other parameters.

The relationship between relative humidity and crushability index

of pre-cooked freeze-dried beef is shown in Table 22. This relationship on the same samples after rehydration is shown in Table 23.

Within the same R.H., the crushability index increased with depth, and this is easily understood since irreversible crushing also increases. Within the same penetration depth, the changes of the crushability index with R.H. were small and difficult to evaluate. The crushability index will be discussed further in the section on the special NLABS foods, and in the chapter on the statistical analysis of data.

The relationship between relative humidity and cohesiveness of pre-cooked freeze-dried beef is shown in Table 24. This relationship on the same samples after rehydration is shown in Table 25. In general, the largest differences were observed between the dry and the rehydrated samples, with cohesiveness much smaller in the dry than in the rehydrated food. Within the dry and rehydrated samples, the effect of relative humidity, as well as the effect of penetration depth, on cohesiveness is small and difficult to evaluate from this Table alone. This effect, together with other results, will be discussed further in connection with the NLABS special foods, and also in the chapter on the Analysis of Variance.

Table 18. Masticometer relative force values of pre-cooked freeze-dried beef in the dry state, after equilibration at different relative humidities, S.I.K. II.³²

Penetration depth %	Force, KR, at relative humidity, %,			
	0	12	23	66
30	537	677	638	720
46	922	921	1045	1103
63	855	936	963	1130
69	1250	1296	1302	1451

Table 19. Masticometer relative force values of pre-cooked freeze-dried beef in the rehydrated state, after equilibration at different relative humidities, S.I.K. II

Penetration depth %	Force, KR, at relative humidity, %,			
	0	12	23	66
26	118	119	129	145
50	245	261	290	309
67	502	500	494	575
78	790	830	762	874

Table 20. Elastic, crushing and total texture behavior of pre-cooked freeze-dried beef in the dry state, after equilibration at different relative humidities, S.I.K. II

	Penetration depth	Instrument values at relative humidity, %,			
	%	0	12	23	66
Elastic	30	49	59	53	63
	46	78	80	89	103
	65	75	90	88	96
	67	112	120	124	140
Crushing	30	90	83	89	89
	46	155	170	192	173
	65	179	198	193	223
	67	251	285	258	279
Total	30	188	201	194	215
	46	263	330	370	380
	65	330	378	368	416
	67	474	524	506	558

Table 21. Elastic, crushing and total texture behavior of pre-cooked freeze-dried beef in the rehydrated state, after equilibration at different relative humidities, S.I.K. II

	Penetration depth %	Instrument values at relative humidities, %,			
		0	12	23	66.
Elastic	26	11	11	12	13
	50	22	25	28	29
	67	44	45	44	52
	78	67	71	63	73
Crushing	26	13	13	15	16
	50	32	38	42	39
	67	72	74	69	82
	78	116	123	114	127
Total	26	35	42	39	42
	50	75	87	98	98
	67	160	164	156	186
	78	250	265	240	274

Table 22. Crushability index of pre-cooked freeze-dried beef in the dry state, after equilibration at different relative humidities, S.I.K. II

Penetration depth %	Instrument values at relative humidity, %,			
	0	12	23	66
30	1.83	1.54	1.79	1.47
46	2.16	2.59	2.36	1.67
65	2.46	2.37	2.38	2.54
67	2.27	2.36	2.13	2.01

Table 23. Crushability index of pre-cooked freeze-dried beef in the rehydrated state, after equilibration at different relative humidities, S.I.K. II

Penetration depth %	Instrument values at relative humidity, %,			
	0	12	23	66
26	1.20	1.16	1.26	1.18
50	1.47	1.57	1.50	1.34
67	1.64	1.80	1.57	1.61
78	1.77	1.79	1.84	1.74

Table 24. Percent cohesiveness of pre-cooked freeze-dried beef in the dry state, after equilibration at different relative humidities, S.I.K. II

Penetration depth %	Instrument values at relative humidity, %, .			
	0	12	23	66
30	52	52	51	56
46	47	44	43	52
65	45	45	44	48
67	51	49	49	53

Table 25. Percent cohesiveness of pre-cooked freeze-dried beef in the rehydrated state, after equilibration at different relative humidities, S.I.K. II

Penetration depth %	Instrument values at relative humidity, %, .			
	0	12	23	66
26	76	70	82	77
50	74	72	74	77
67	71	73	74	74
78	69	71	69	71

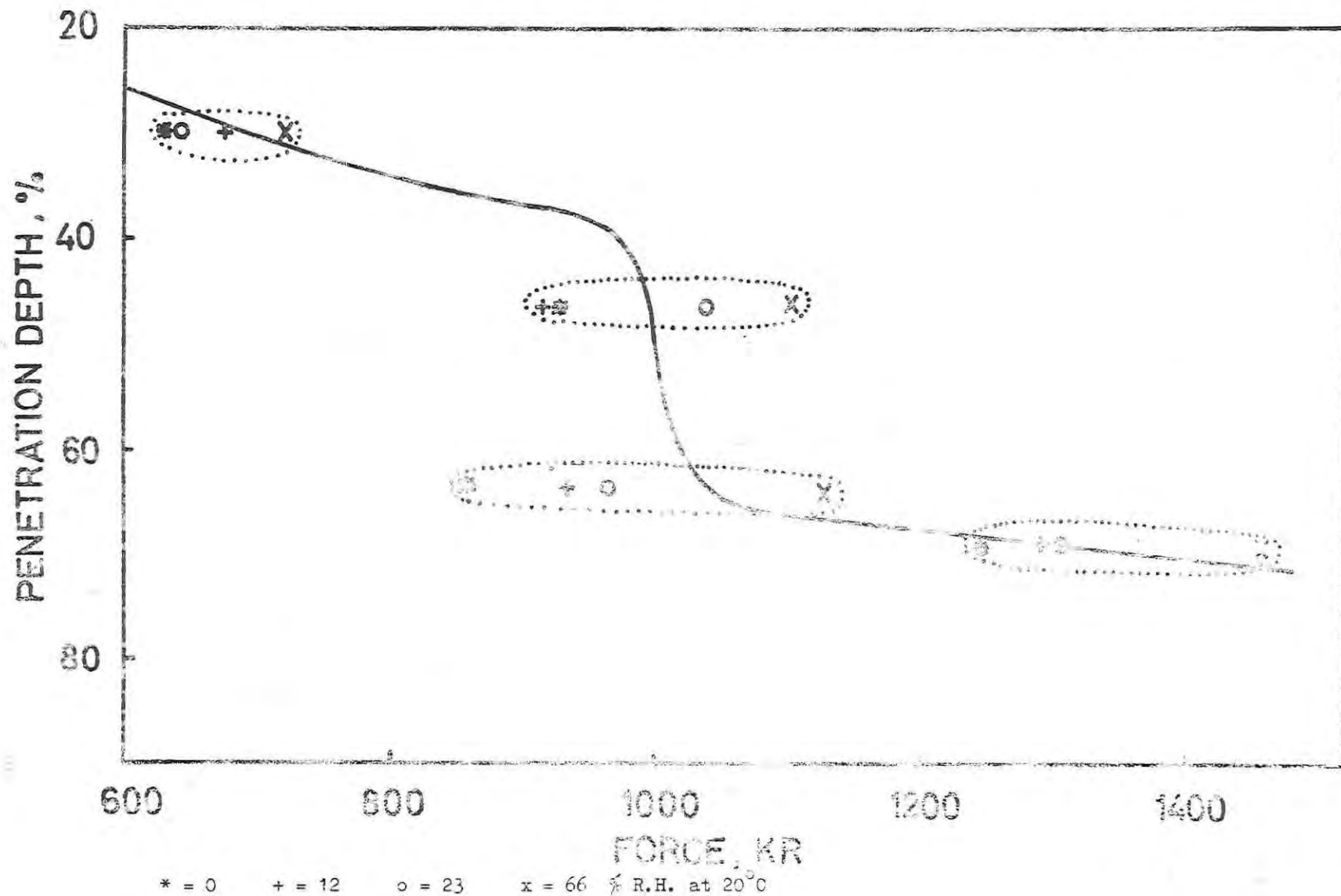
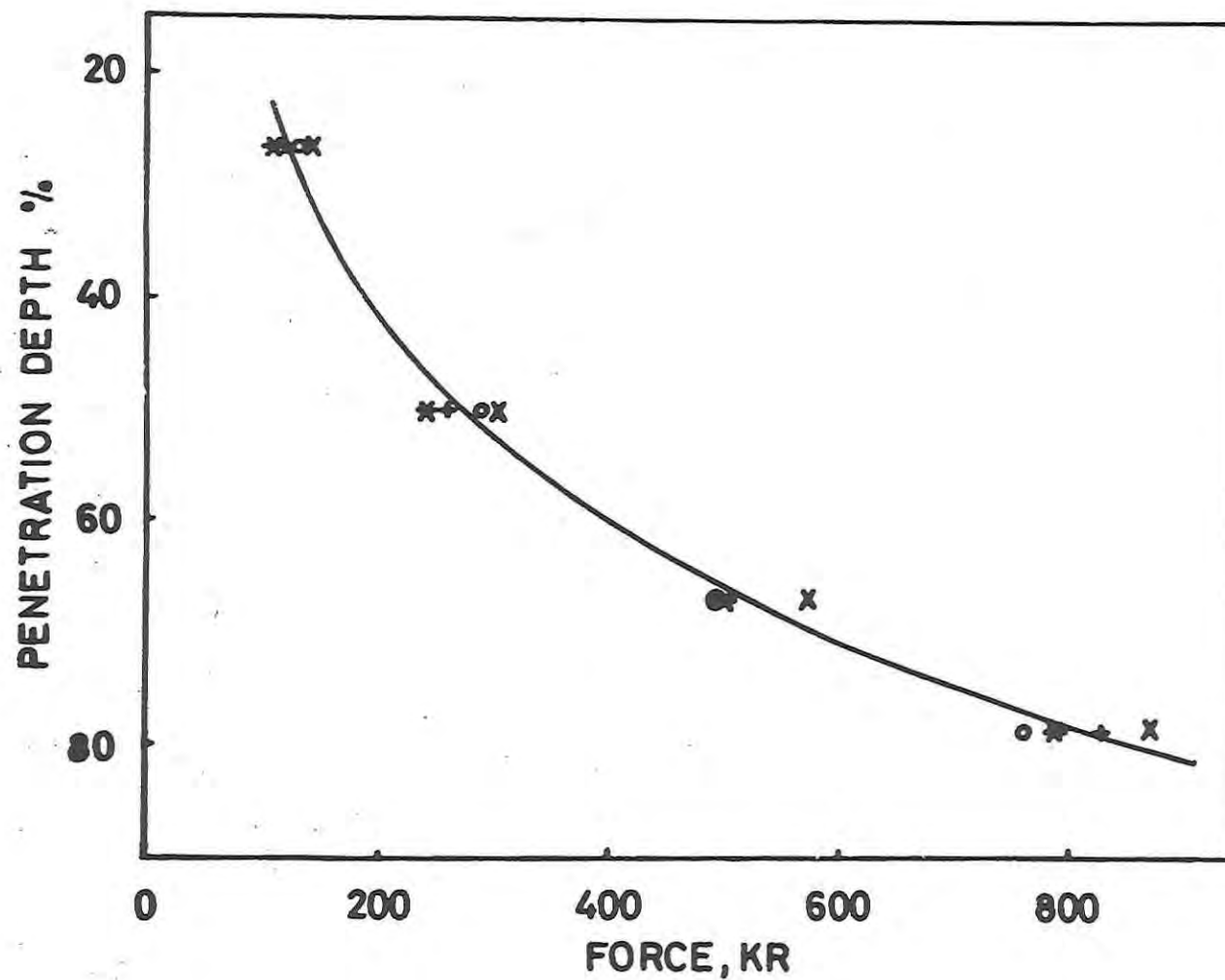


Fig. 22. Plot of values of instrument force vs. penetration depth in pre-cooked freeze-dried beef in the dry state, after equilibration at different relative humidities S.I.K. II.



* = 0 + = 12 o = 23 x = 66 % R.H. at 20°C

Fig. 23. Plot of values of instrument force vs. penetration depth in pre-cooked freeze-dried beef in the rehydrated state, after equilibration at different relative humidities, S.I.K. II.

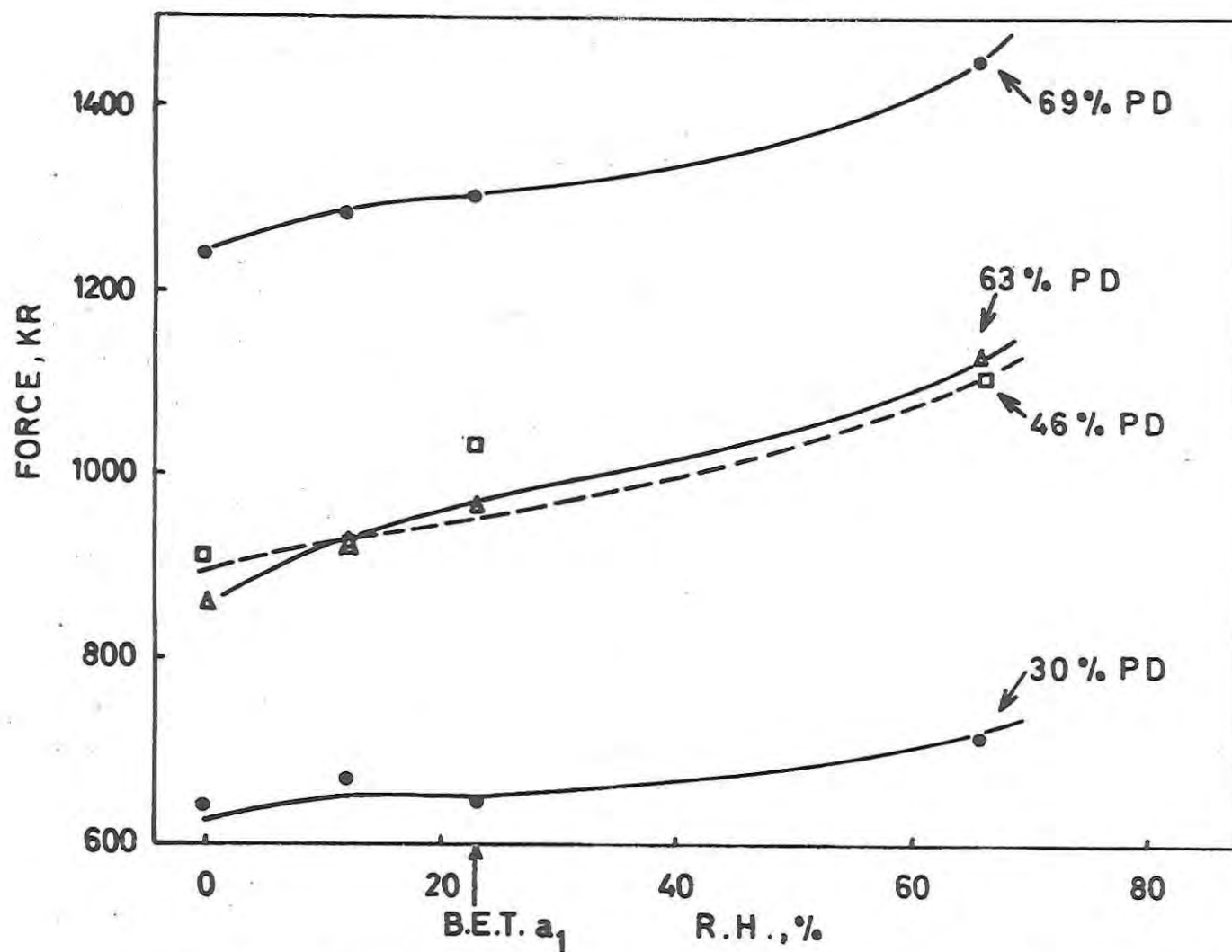


Fig. 24. Plot of values of % relative humidity at 20°C vs. values of instrument force of penetration in pre-cooked freeze-dried beef in the dry state, S.I.K. II.

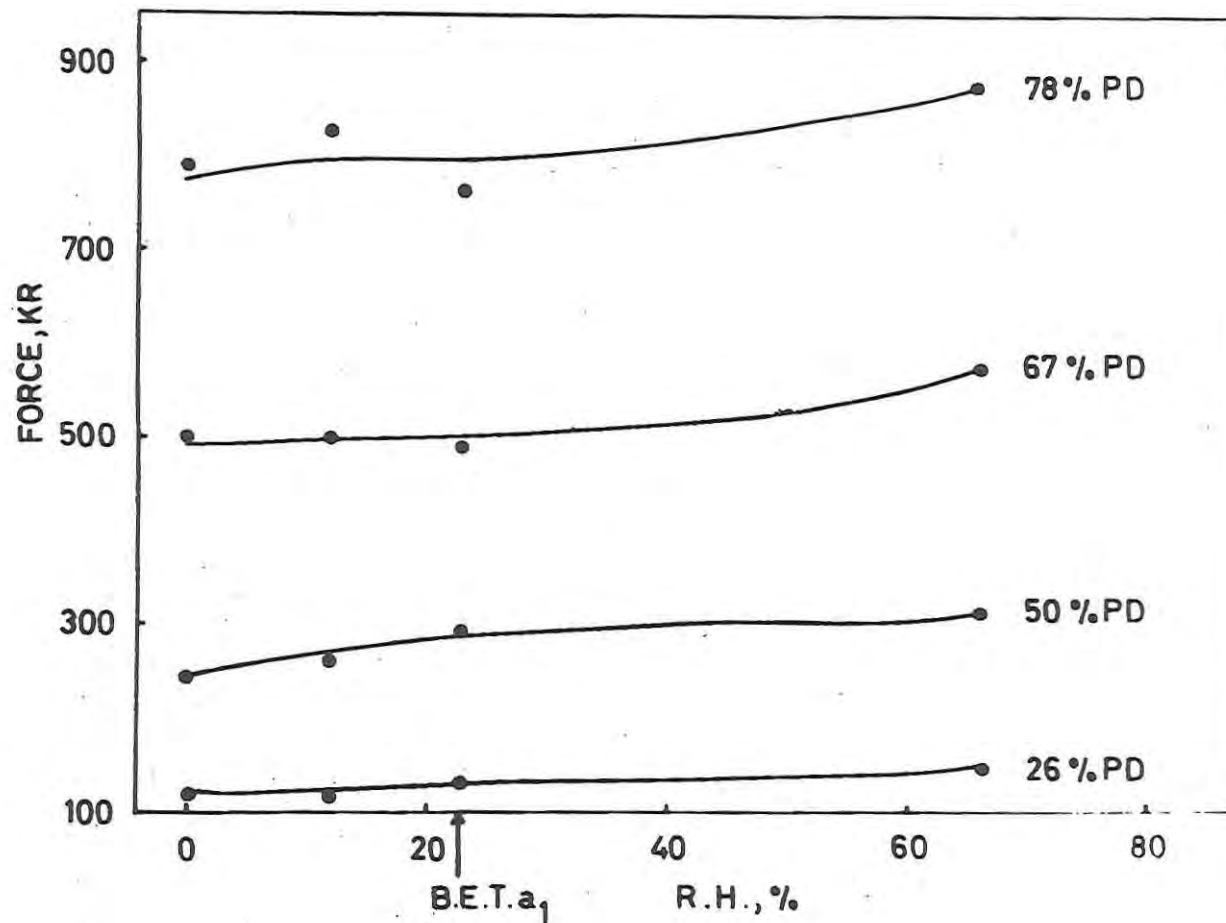
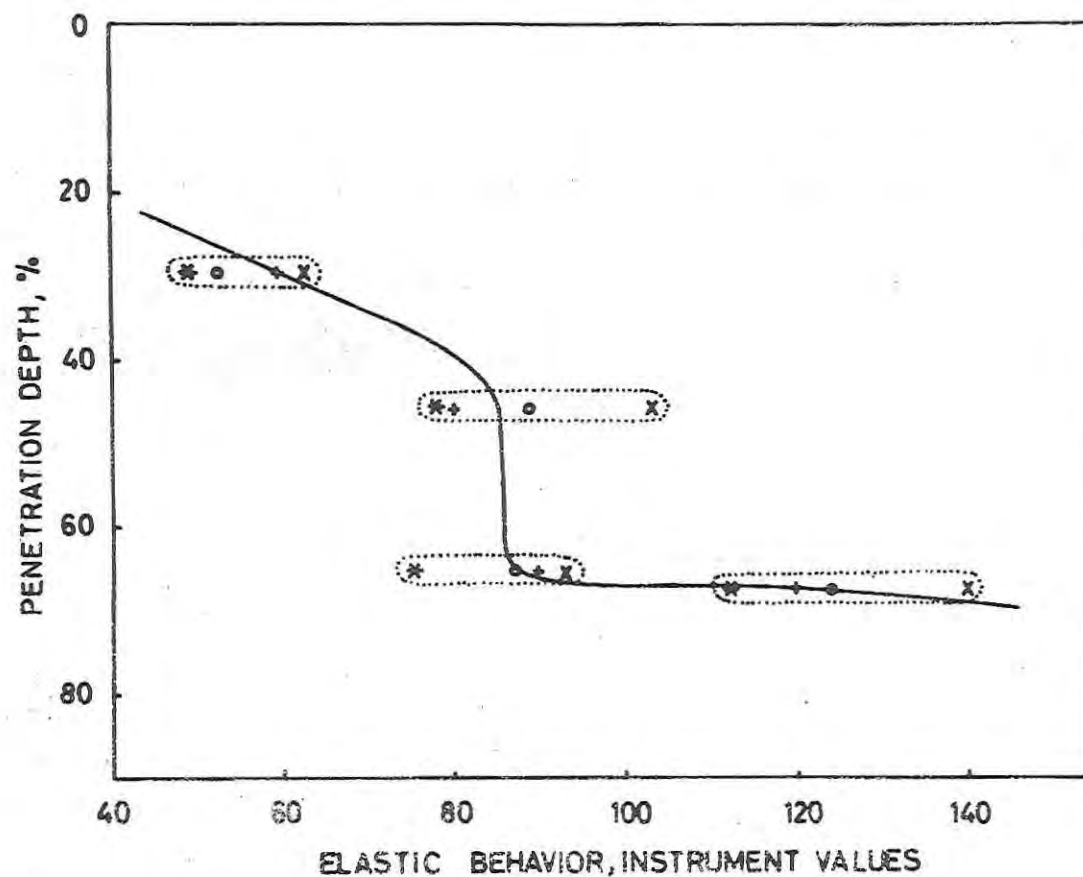
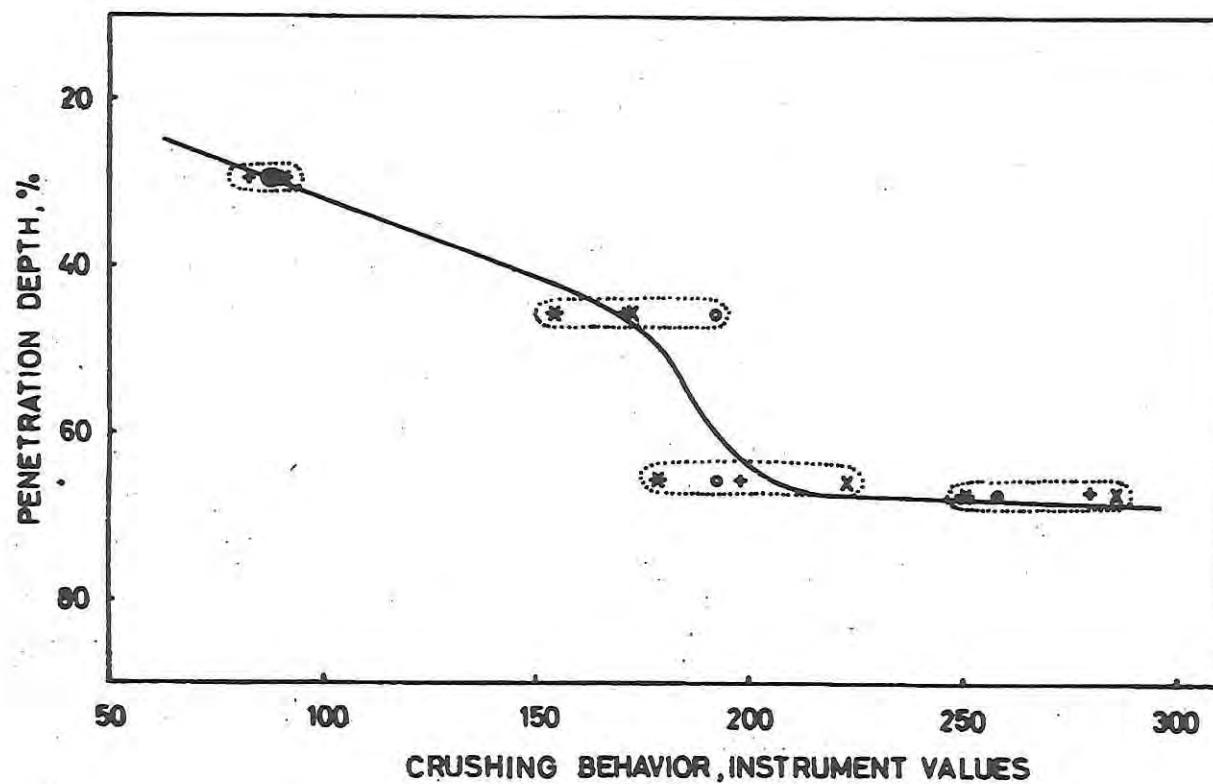


Fig. 25. Plot of values of % relative humidity at 20°C vs. values of instrument force of penetration in pre-cooked freeze-dried beef in the rehydrated state, S.I.K. II.



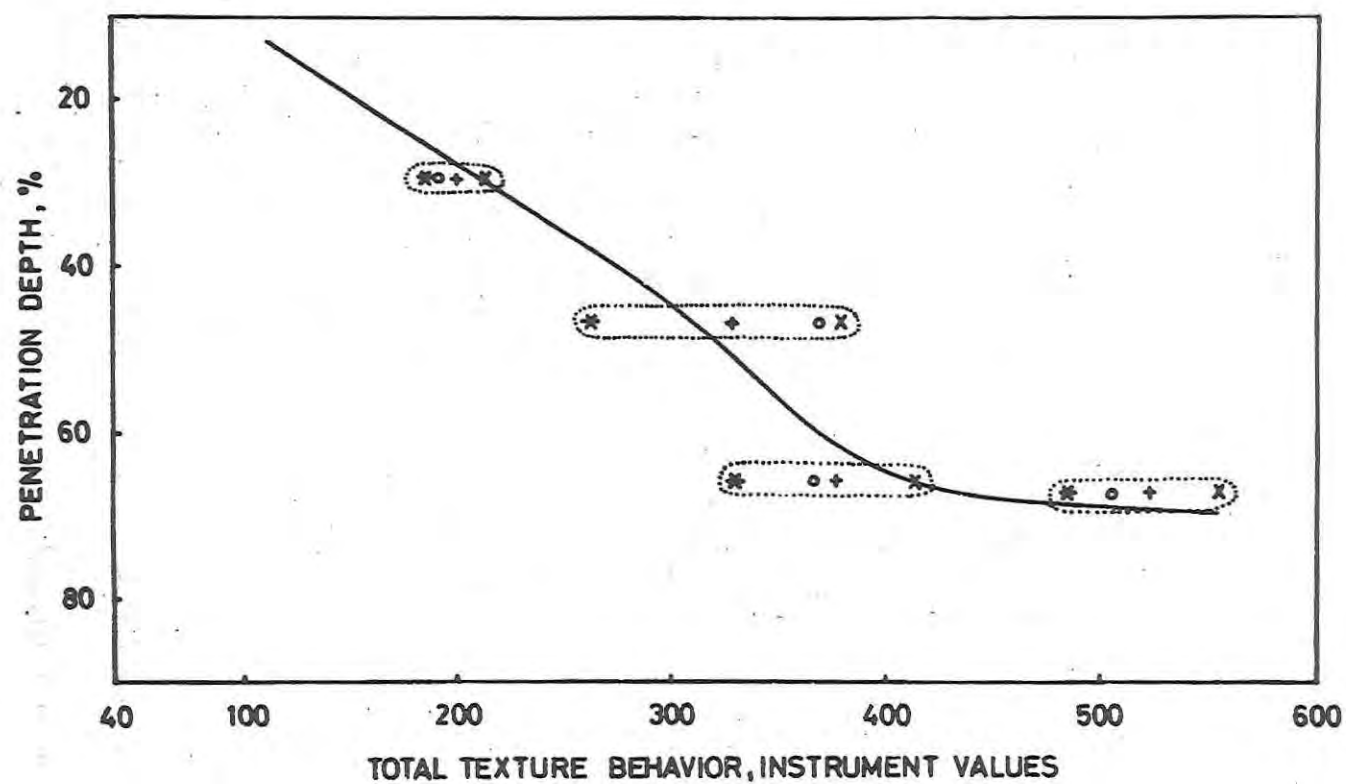
* = 0 + = 12 o = 23 x = 66 % R.H. at 20°C.

Fig. 26. Plot of values of elastic behavior of pre-cooked freeze-dried beef in the dry state, after equilibration at different relative humidities, vs. penetration depth, S.I.K. II.



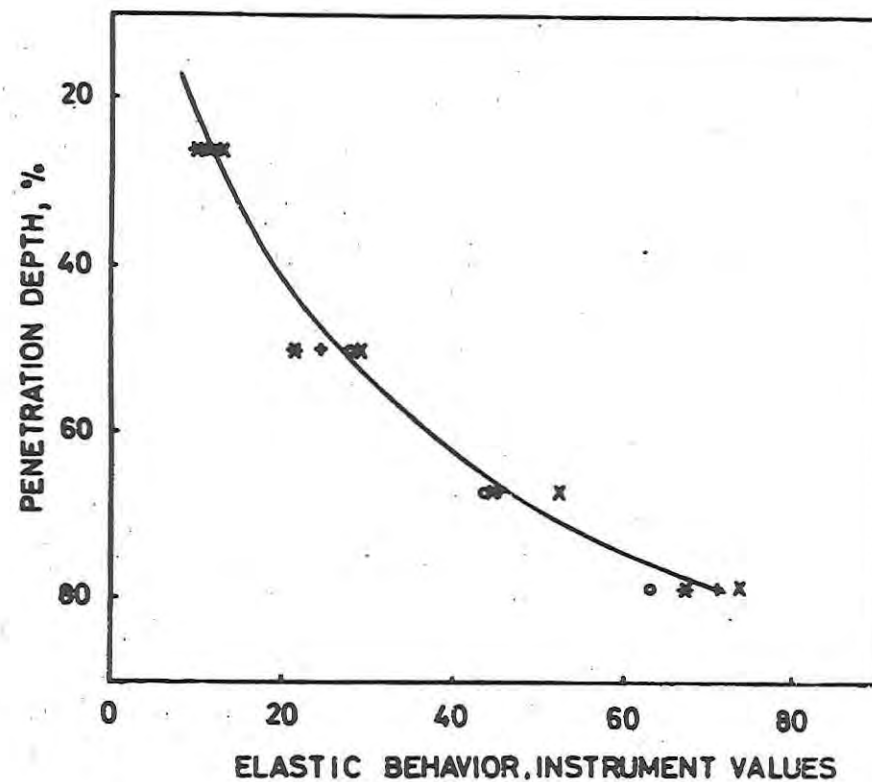
* = 0 + = 12 o = 23 x = 66 % R.H. at 20°C.

Fig. 27. Plot of values of crushing behavior of pre-cooked freeze-dried beef in the dry state, after equilibration at different relative humidities, vs. penetration depth, S.I.K. II.



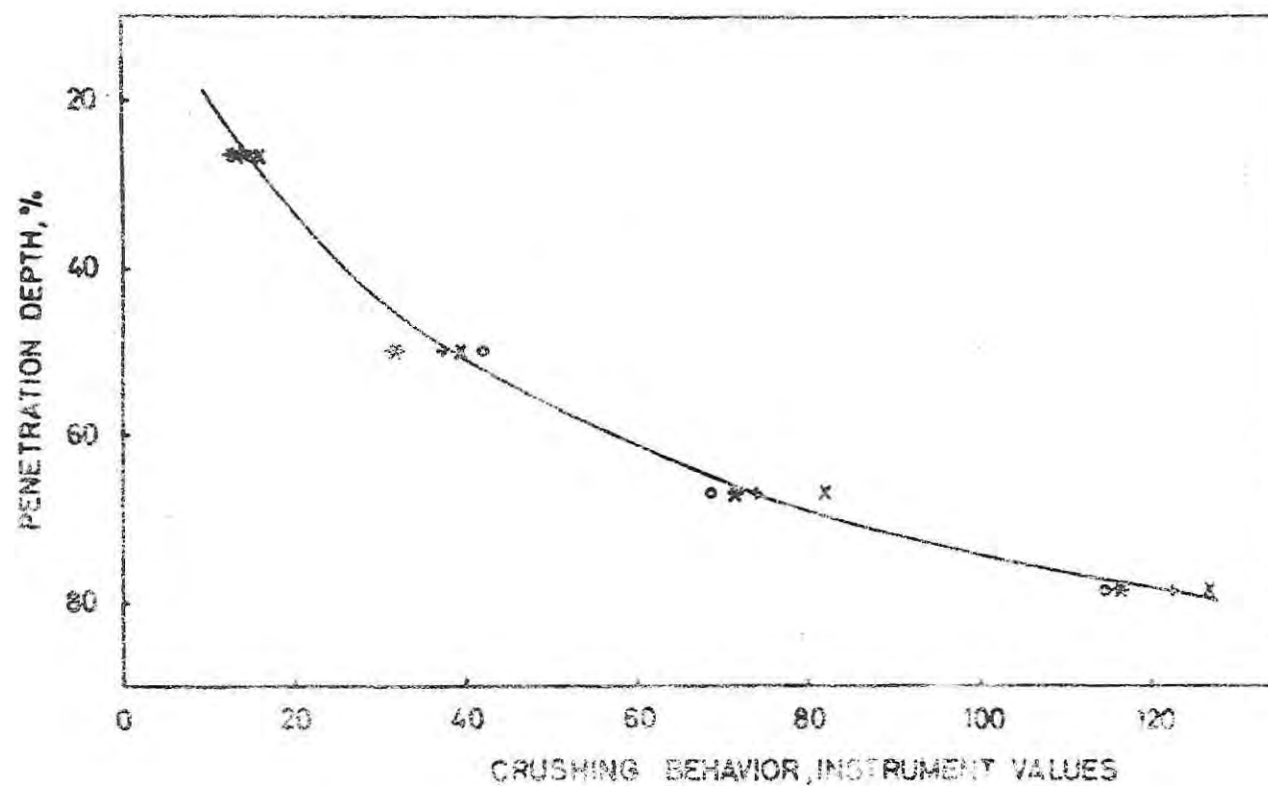
* = 0 + = 12 o = 23 x = 66 % R.H. at 20°C.

Fig. 28. Plot of values of total texture behavior of pre-cooked freeze-dried beef in the dry state, after equilibration at different relative humidities, vs. penetration depth, S.I.K. II.



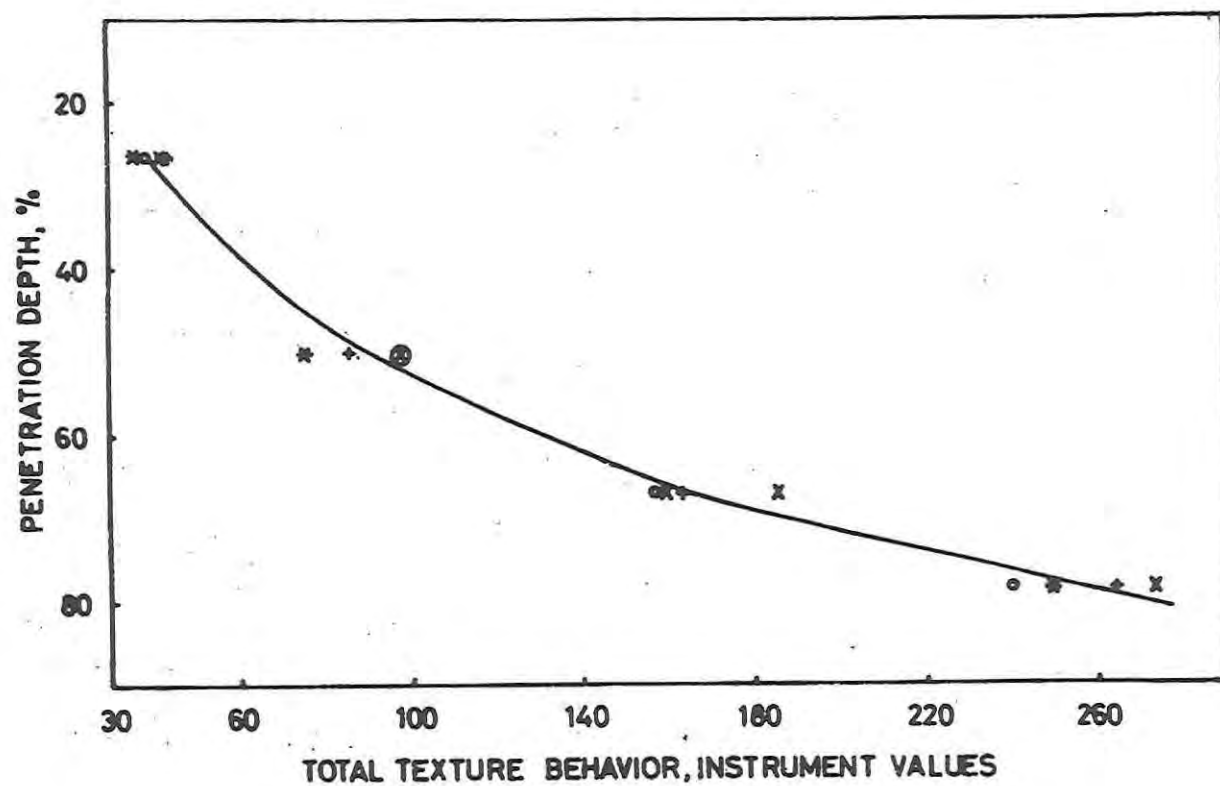
* = 0 + = 12 o = 23 x = 66 % R.H. at 20°C.

Fig. 29. Plot of values of elastic behavior of pre-cooked freeze-dried beef in the rehydrated state, after equilibration at different relative humidities, vs. penetration depth, S.I.K. II.



* = 0 + = 12 o = 23 x = 66 % R.H. at 20°C.

Fig.30. Plot of values of crushing behavior of pre-cooked freeze-dried beef in the rehydrated state, after equilibration at different relative humidities, vs. penetration depth, S.I.K. II.



* = 0 + = 12 o = 23 x = 66 % R.H. at 20°C.

Fig. 31. Plot of values of total texture behavior of pre-cooked freeze-dried beef in the rehydrated state, after equilibration at different relative humidities, vs. penetration depth, S.I.K. II.

REHYDRATION BEHAVIOR

The rehydration behavior in terms of the coefficient of weight restoration and of the total percent moisture on non-fat dry basis is shown in Table 26. Each number of the I and II series represents the mean value of 16 different samples and each number of the III and IV series represents the mean value of 8 different samples.

The coefficient of weight restoration has been used frequently in the literature, and it is included here for reference purposes. Since the moisture in the sample increases with increasing relative humidity, the coefficient of weight restoration decreases, and any real differences in sorption characteristics due to alterations of the dry material are not shown explicitly. For this purpose, the total % moisture on N.F.D. basis was examined.

Meat from the 2-year-old animals I and III absorbed less % total water on non-fat basis than meat from the 5-year-old animals II and IV. No definite explanation for this phenomenon is suggested. It is possible that the genetic make-up of the animals was different in this respect, or that the meat of the young animals was more adversely affected by the storage conditions at 20°C. The effect of the relative humidity on the total moisture is less clear. Samples I, III and IV absorbed less and sample II more total moisture at 66 than at 0 % R.H., but the differences are too small to draw any definite conclusions. Only small differences were also observed among different relative humidities within each sample series I, II, III and IV.

Table 26. Rehydration behavior of pre-cooked freeze-dried beef.
S.I.K. I - IV

S.I.K. Series	R.H. %	Coefficient of weight restoration(x100)	total % moisture, N.F.D.	
I	0	230	152	
	12	222	154	
	23	217	152	
	66	199	<u>143</u>	150
II	0	250	180	
	12	245	184	
	23	236	177	
	66	230	<u>187</u>	182
III	0	259	167	
	12	255	173	
	23	244	181	
	66	228	<u>163</u>	171
IV	0	254	183	
	12	236	168	
	23	236	173	
	66	221	<u>172</u>	174

SPECIAL FOODS, NLABS SAMPLE SERIES

Masticometer curves for beef, cheese, and chicken sandwich are shown in Fig. 32, 33 and 34; respectively. Certain variations of the shape of these curves were experienced among different samples of the same food. The sequence of recording is from left to right: the small peaks at equal intervals (marked p) represent the time of the uppermost position of the punch, whereas the dashed line within the first experimental peak represents the time of the lowest position of the punch (cf., Figs. 9, 11). The curves are unusual in certain respects. In the beef and cheese sandwiches, as the punch penetrated the first bread layer the force reached a maximum, which is shown by the highest of the two peaks of the first experimental curve. As the punch penetrated the second bread, there was again an increase of force, which this time was smaller, due to the already weakened or fractured structure of the second bread under the pressure of the punch on the first bread and on the meat layer. In the curve for chicken sandwich shown here (Fig. 34), there were larger variations of force as the punch penetrated layers of different hardness within a very crumbly structure. This crumbliness or irreversible crushing is indicated by the asymmetry of the first experimental peak about the vertical dashed line, which line, as explained previously, corresponds to the lowest position of the punch.

The effect of relative humidity on the hardness (Force, KR) crushability index, and % cohesiveness of dehydrated special foods is shown in Table 27 and Figs. 35 and 36. The excessive crumbliness of these foods is indicated by the high crushability index and the low % cohesiveness (cf., Tables 22 and 24). In general, as the relative humidity increased, the force and the cohesiveness increased, and the crushability index decreased. This is shown more dramatically in the dehydrated chicken sandwich in Fig. 36. The toughening effect of increasing moisture levels is exerted through a decrease of the contribution of the irreversible crush-

ing parameters to the total textural quality of the food. At the same time, the bridges, formed through chemical interactions at the higher moisture levels, increase the strength of the internal bonds which bind the individual parts into the whole structure, and as a result the cohesiveness of the system increases. In this way, qualitatively, the special dry food begins behaving like a conventional rehydrated product.

In general, the effect of the relative humidity on the hardness, crushability index and cohesiveness was much more pronounced in the NLABS dehydrated foods than in the S.I.K. dehydrated meat series. (cf., Tables 18, 22, 24 and 27). This is probably due to the much higher content of carbohydrate material (bread etc) in the NLABS than in the S.I.K. foods. The textural parameters of carbohydrate materials are probably affected by moisture changes to a much greater extent than the textural parameters of protein materials. These concepts need to be further investigated before final conclusions can be made.

Table 27. Values of Masticometer parameters in relation to relative humidity in dehydrated special foods (punch diameter 5 mm).

Food and % PD	Texture parameter	Values at relative humidity, %, <div>0 12 23 66</div>			
		0	12	23	66
<u>Beef sandwich,</u> <u>80% PD</u>	Force, KR	692	796	865	1135
	Crushability index	9.51	8.79	9.63	7.51
	Cohesiveness, %	20	25	23	33
<u>Chicken sand-</u> <u>wich, 80% PD</u>	Force, KR	695	799	1070	1277
	Crushability index	7.15	5.85	6.23	4.63
	Cohesiveness, %	41	43	34	43
<u>Cheese sand-</u> <u>wich, 80% PD</u>	Force, KR	817	1040	1193	1722
	Crushability index	22.1	19.9	16.8	7.6
	Cohesiveness, %	13	14	16	27
<u>Chicken bites</u> <u>59% PD</u>	Force, KR	505	552	677	787
	Crushability index	5.71	5.38	4.54	3.26
	Cohesiveness, %	24	25	28	43

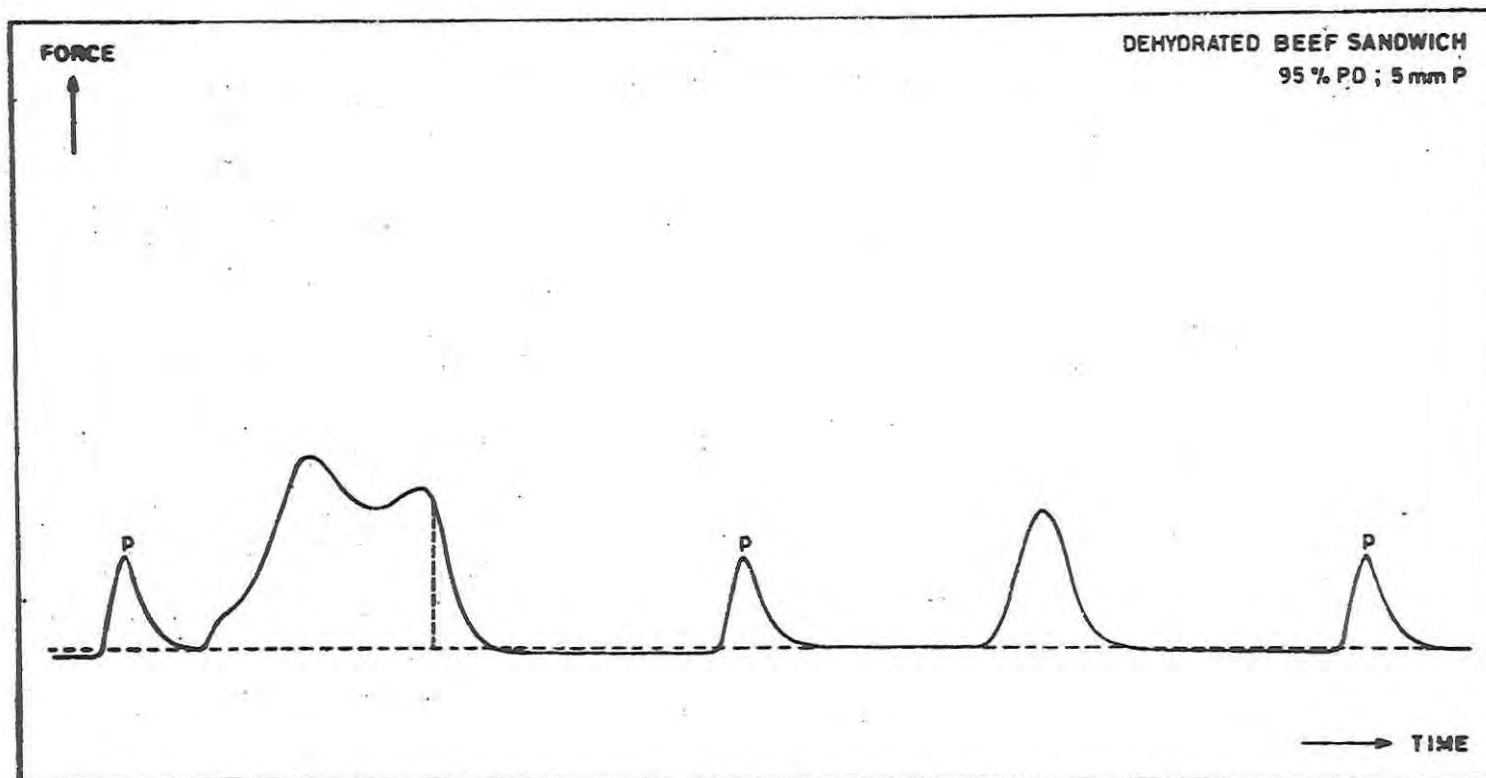


Fig. 32. Masticometer curve for dehydrated beef sandwich. p = uppermost position of punch

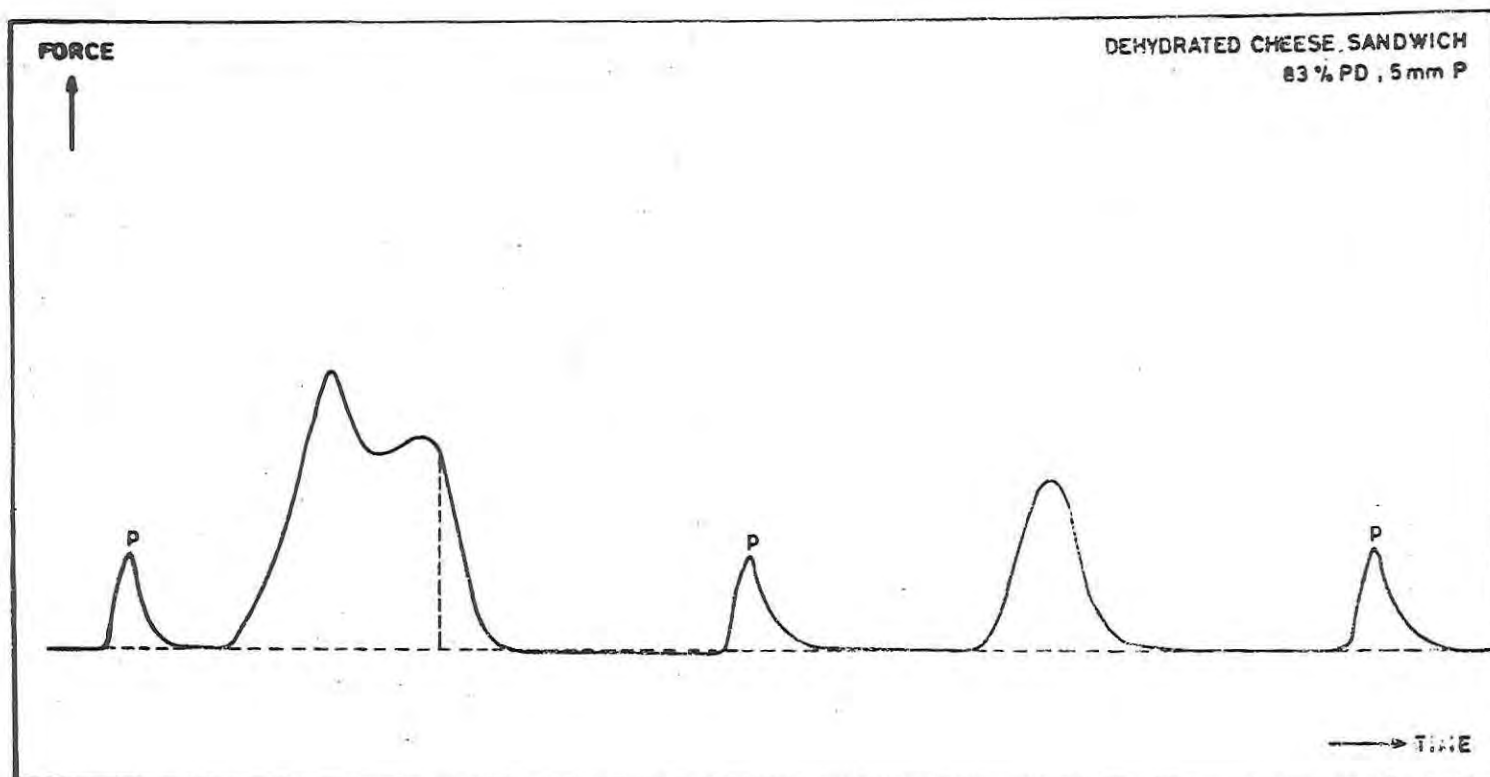


Fig. 33. Masticometer curve for dehydrated cheese sandwich. p = uppermost position of punch

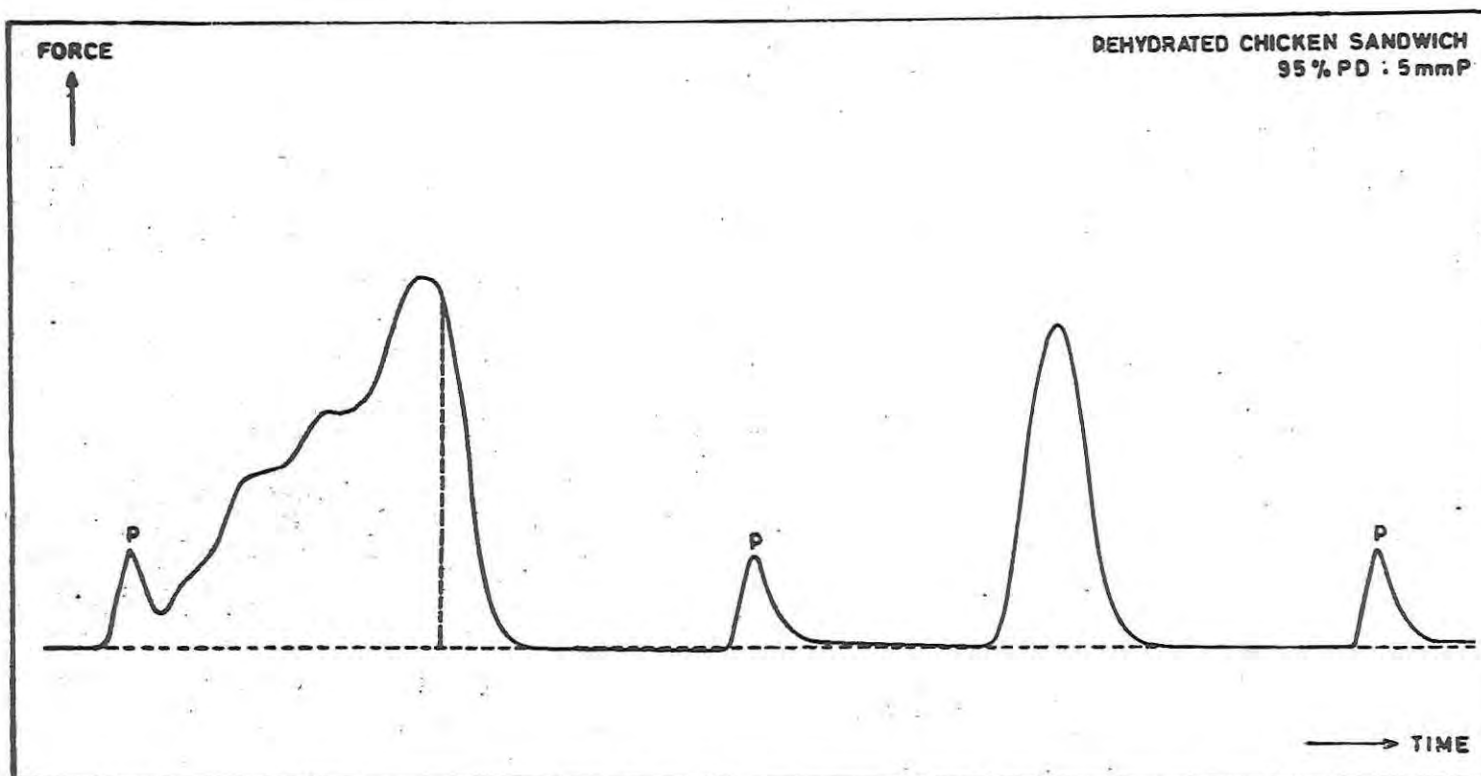


Fig. 34. Masticometer curve for dehydrated chicken sandwich. p = uppermost position of punch

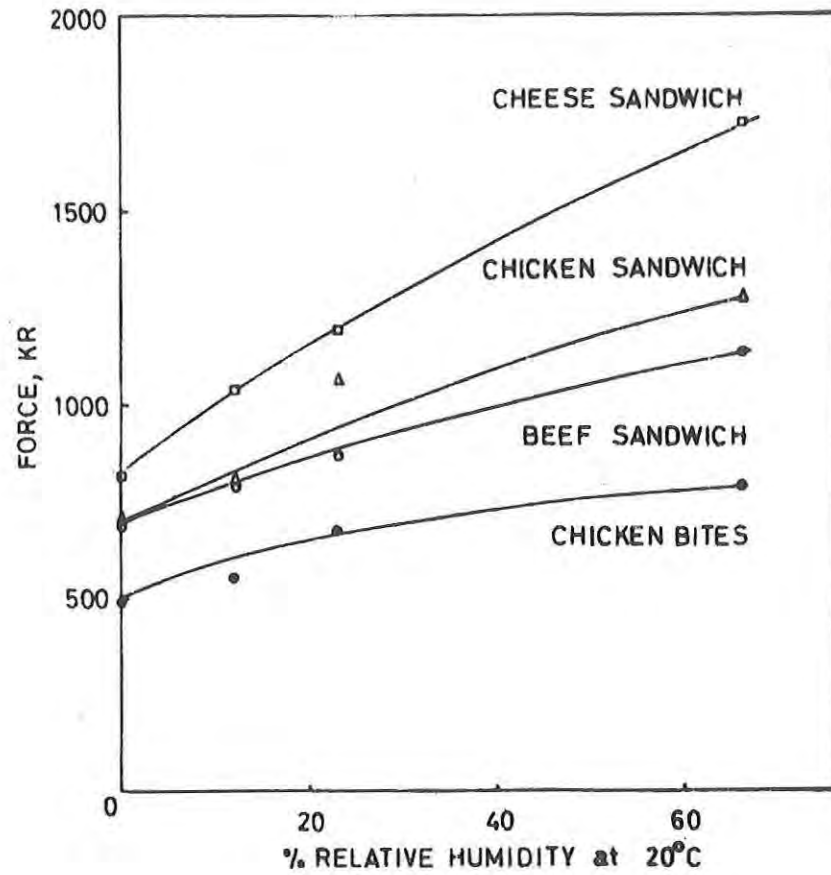


Fig. 35. Plot of values of instrument force of penetration vs. relative humidity in dehydrated special foods, NLABS.

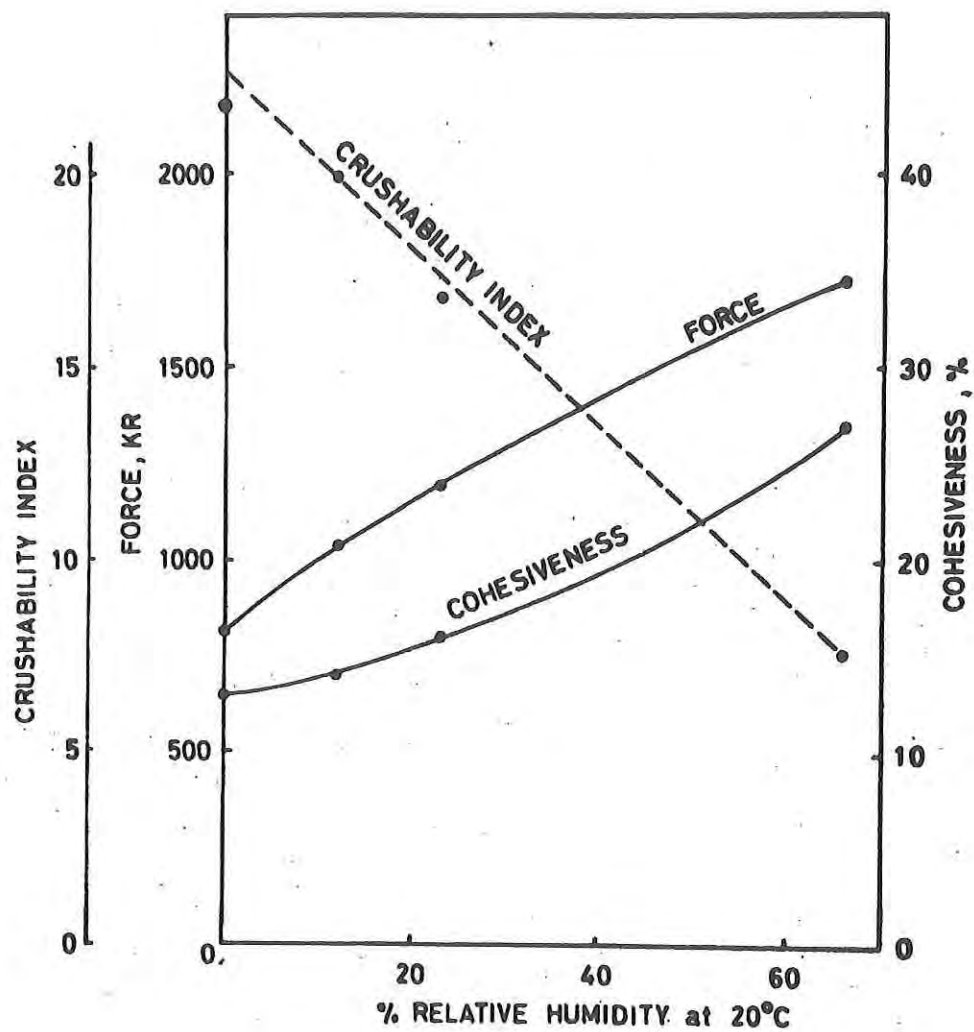


Fig. 36. Plot of values of Masticometer texture parameters vs. relative humidity in dehydrated cheese sandwich, NLABS.

PHYSICO-CHEMICAL VARIABLES

General Characteristics

The values for the B.E.T. monomolecular layer of water and for the corresponding surface area at the different temperatures of the experiments are presented in Table 28. At 20°C, which was the temperature used for the instrument and texture measurements, the chicken sandwich exhibited the largest and the melba toast the smallest monolayer values and surface area. The S.I.K. food sample series I to IV exhibited closely similar monolayer values, with a mean of 3.39 %. This value is in agreement with other reported work (89).

The partial molal thermodynamic data for water vapor sorption at 20°C are presented in Tables (29-31). These data are also plotted in Figs. 37-45. The integral thermodynamic values for the same foods are shown in Tables 32 and 33.

Of all thermodynamic values, the partial molal enthalpy change $-\bar{\Delta}H$ showed the greatest differences among foods. In most cases, this change was highest at zero relative humidity, where the affinity of water for the dry material is greatest. This may be due to sorption (possibly by hydrogen bond formation) on polar groups of the protein side chains, as proposed first by Pauling (78) and then by others (18, 22). As the relative humidity increased, the enthalpy change first dropped rapidly and then gradually, approaching a few calories per mole at a point close to the value of two B.E.T. layers of water. However, in chicken bites (Fig. 44), the value first rose up to about 23% R.H., and then decreased. In most cases, there was some residual heat of adsorption at high relative humidities, and the enthalpy curve approached the abscissa asymptotically. Davis and McLaren (18) attributed this to the presence of a spectrum of sorption and chemisorption energies, with the sites of chemisorption becoming completely satiated only in solution. The presence of net heat of adsorption beyond the first layer of

water violates the assumption of the B.E.T. theory that the heat of adsorption beyond the first layer is equal to the heat of condensation in pure water. This violation of the B.E.T. theory has also been observed in other work (22).

In a previous paper, Kapsalis et al. (51) discussed the presence of residual heat of adsorption at higher relative humidities on the basis of the "zipper mechanism" of water sorption in mucopolysaccharides, as proposed recently by Ehrlich and Bettelheim (29). These authors consider two kinds of sorption sites: the polar groups on the surface which are readily available for water sorption, and the inner, tightly hydrogen-bonded polar sites which are less readily available. The primary sorption takes place on free polar sites; subsequent swelling opens up previously bonded, and therefore inaccessible, sites for further sorption. Thus, through what may be visualized as a zipper mechanism, the water molecules penetrate into the matrix, first occupying free sites, and subsequently breaking up existing hydrogen bonds between polymer chains and establishing new ones with the sorbent. (In a reverse fashion, the desorption process will first break up hydrogen bonds between water and polar groups of the polymer, and it will next establish new ones between the polymer chains). It is possible that the completion of a first layer of water on the inner, tightly-bonded sites occurs simultaneously with the formation of a second layer on the outer, free polar sites. On the basis of these considerations, the residual heat of adsorption at higher relative humidities may be attributed to delayed sorption on inner sites, as the latter become available through the zipper mechanism.

As in the case of the heat of adsorption, the partial molal entropy change $-\Delta\bar{S}$ was highest at low relative vapor pressures. Among the foods studied, the S.I.K. series I samples exhibited the highest, and the beef sandwich the lowest change. The negative value is due to the decrease in randomness of the system

when water molecules from the vapor phase are adsorbed on the surface. High negative entropy values may be due to chemisorption as postulated by Pauling (78). As the relative vapor pressure increases, the additional water molecules adsorbed on the surface have greater freedom of movement than the tightly held molecules, and the entropy change becomes less negative. It is interesting to note the positive entropy changes observed in the S.I.K. series I-IV, and in the NLABS' chicken bites, beef sandwich and melba toast. Such positive entropy gains were previously observed in salmin, lyophilized lactalbumin, and crystalline egg albumin by Davis and McLaren (18). These authors and also Bull (9) attributed them to configurational changes of the sorbing surface, i.e., to a local solubilization or incipient solution with a resultant positive entropy of mixing. In melba toast and beef sandwich, this occurred at as low relative humidity as 20 and 30% respectively, i.e., long before these materials were dissolved, and this is similar to the behavior of salmin at 30% relative humidity (18). Supporting these concepts are the experiments of King (53), who attributed the increased dielectric constant of gelatin and the decreased rigidity of wool, accompanying the sorption of water, to increased rotation of polar groups in polypeptide chains.

The partial molal free energy change - $\overline{\Delta G}$ at 20°C, since it depends on the temperature and the relative humidity was of course the same in all foods.

Within the time schedule of this Report, the values of the Fugassi constants for the experimental data of this study were not yet available from the Data Analysis Branch of the U.S. Army Natick Laboratories. These values will be included in any future publications of results from this Report.

Relationships with rheological parameters

In this Report, the moisture sorption isotherm was the basis for the calculation of the physico-chemical variables and for the study of the relationships between the different hygroscopic equilibrium values and the rheological parameters of the foods. For this reason, the general characteristics discussed above, apply also when the different thermodynamic values are plotted against the Masticometer parameters.

There is a great deal of information stored in the experimental results of this study, for present and future use. For the sake of this discussion, only the relative Masticometer force will be examined. The results are shown in Table 34 and Figs. 46-48.

At higher $-\overline{\Delta H}$ values, the affinity of moisture for the sorptive sites is greatest (56). The water molecules are part of the original structure and, interposed between adjacent polymer chains, they exert a favorable effect with regard to tenderness. This results in lower values of force of penetration. As the $-\overline{\Delta H}$ value decreases (approaching the heat of condensation of water at a relative humidity corresponding to the completion of two B.E.T. monolayers), the water molecules are less firmly bound to the protein structure, and they become more readily available for cross-linking reactions which result in higher force values, i.e., in a tougher food product (29, 70).

The high relative entropy values may be due to water which is hydrogen bonded on polar sites of the protein. The "immobility" of water within the B.E.T. monolayer has been confirmed by nuclear magnetic resonance studies on starch (93), and it offers another way of considering the above mentioned beneficial effect of water within the meat matrix.

The $-\overline{\Delta G}$ value, by sake of its derivation and meaning, reflects the composite effect of the changes in the partial

molal enthalpy and entropy. At higher $-\overline{\Delta G}$ values, the spontaneity of the sorption process is greatest and the force of penetration smallest. The reverse is the case when the $-\overline{\Delta G}$ value decreases.

Table 28. B.E.T. monomolecular layer of water a_1 , and the corresponding surface area in dehydrated foods.

Food	Values at a temperature of									
	4°C		10°C		15°C		20°C		30°C	
	a_1^*	Area**	a_1^*	Area**	a_1^*	Area**	a_1^*	Area**	a_1^*	Area**
SIK I	3.55	128	3.48	126	3.41	123	3.66	132	3.86	139
SIK II	4.61	166	3.35	121	3.53	127	3.29	119	3.60	130
SIK III	4.02	145	3.82	138	3.13	113	3.18	115	3.31	119
SIK IV	4.30	155	3.82	138	3.79	137	3.45	125	3.32	120
Beef sandwich	4.10	148	3.70	134	3.54	128	3.51	127	2.96	107
Chicken sandwich	4.30	155	3.83	138	3.69	133	3.88	140	3.02	109
Cheese sandwich	4.31	156	3.17	114	3.06	110	2.89	104	2.69	97
Chicken bites	4.59	166	4.74	171	3.84	138	4.39	158	3.18	115
Melba toast	2.84	103	2.31	83	2.42	87	2.14	77	1.75	63

* g water/100g N.F.D. matter

** m²/g N.F.D. matter

Table 29. Partial molal thermodynamic data for water vapor sorption in dehydrated beef at 20°C. S.I.K. I - IV.

R.H. %	S.I.K. I				S.I.K. II			S.I.K. III			S.I.K. IV		
	$-\Delta G^{(1)}$	$n^{(2)}$	$-\overline{\Delta H}^{(1)}$	$-\overline{\Delta S}^{(3)}$	$n^{(2)}$	$-\overline{\Delta H}^{(1)}$	$-\overline{\Delta S}^{(3)}$	$n^{(2)}$	$-\overline{\Delta H}^{(1)}$	$-\overline{\Delta S}^{(3)}$	$n^{(2)}$	$-\overline{\Delta H}^{(1)}$	$-\overline{\Delta S}^{(3)}$
0	∞	0.013	12100	-	0.015	7720	-	0.010	9900	-	0.012	9940	-
5	1743	0.084	10350	29.4	0.093	6660	16.8	0.094	8750	23.9	0.089	9439	26.2
11	1285	0.139	7600	21.6	0.142	5850	15.6	0.145	7130	19.9	0.134	8300	23.9
23	855	0.206	4600	12.8	0.196	4850	13.6	0.195	4460	12.4	0.191	5470	15.8
50	403	0.388	510	-0.44	0.380	970	1.94	0.391	480	0.26	0.396	400	0
64	260	0.584	290	-0.01	0.580	0	-0.89	0.580	80	-0.61	0.580	0	-0.89

(1) = cal/mole

(2) = moles water/100g N.F.D. matter

(3) = cal/mole/deg

Table 30. Partial molal thermodynamic data for water vapor sorption in dehydrated foods at 20°C. NLABS 3 special foods.

R.H. %	$-\overline{\Delta G}^{(1)}$	Chicken bites			Chicken sandwich			Cheese sandwich		
		$n^{(2)}$	$-\overline{\Delta H}^{(1)}$	$-\overline{\Delta S}^{(3)}$	$n^{(2)}$	$-\overline{\Delta H}^{(1)}$	$-\overline{\Delta S}^{(3)}$	$n^{(1)}$	$-\overline{\Delta H}^{(1)}$	$-\overline{\Delta S}^{(3)}$
0	∞	0.031	5670	-	0.015	9200	-	0.015	8950	-
5	1743	0.145	6800	17.3	0.121	8250	22.2	0.089	8100	21.7
11	1285	0.193	7340	20.7	0.188	7400	20.9	0.146	7030	19.6
23	855	0.244	4600	12.8	0.240	4650	13.0	0.185	5850	17.0
50	403	0.529	0	-1.38	0.423	1050	2.20	0.430	660	0.91
64	260	0.966	0	-0.89	0.625	320	0.20	0.770	390	0.44

(1) cal/mole

(2) moles water/100g N.F.D. matter

(3) cal/mole/deg

Table 31. Partial molal thermodynamic data for water vapor sorption in dehydrated foods at 20°C,
NLABS 2 special foods.

R.H. %	$-\overline{\Delta G}^{(1)}$	Beef sandwich			Melba Toast		
		$n^{(2)}$	$-\overline{\Delta H}^{(1)}$	$-\overline{\Delta S}^{(3)}$	$n^{(2)}$	$-\overline{\Delta H}^{(1)}$	$-\overline{\Delta S}^{(3)}$
0		0.016	2630	-	0.020	8000	-
5	1743	0.079	2420	2.31	0.084	7350	19.1
11	1285	0.134	2230	3.22	0.116	6840	19.0
23	855	0.194	1870	3.46	0.138	6200	18.2
50	403	0.432	280	-0.42	0.334	C	-1.38
64	260	0.712	60	-0.68	0.596	0	-0.89

(1) = cal/mole

(2) = moles water/100g N.F.D. matter

(3) = cal/mole/deg

Table 32. Integral net heats, net entropies and net free energies of water vapor sorption in pre-cooked freeze-dried beef, S.I.K. I - IV

R.H. %	S.I.K. I			S.I.K. II			S.I.K. III			S.I.K. IV		
	$-\Delta H$	$-\Delta S$	$-\Delta G 20^{\circ}C$	$-\Delta H$	$-\Delta S$	$-\Delta G 20^{\circ}C$	$-\Delta H$	$-\Delta S$	$-\Delta G 20^{\circ}C$	$-\Delta H$	$-\Delta S$	$-\Delta G 20^{\circ}C$
10	1320	4.01	145	884	2.57	131	1120	3.35	138	900	2.61	135
20	1710	5.13	207	1150	3.28	189	1450	4.28	196	1500	4.42	205
40	2120	6.20	303	1562	4.39	276	1880	5.42	286	2010	5.83	302
60	2260	6.36	397	1783	4.80	377	1940	5.34	376	2100	5.84	389
80	2300	6.10	513	1800	4.45	496	1960	5.00	495	2100	5.43	509
100	2300	5.40	718	1800	3.78	693	1960	4.22	724	2100	4.67	732

$-\Delta H = \text{cal/100g N.F.D.}$

$-\Delta S = \text{cal/100g N.F.D./deg}$

$-\Delta G = \text{cal/100g N.F.D.}$

Table 33. Integral net heats, net entropies and net free energies of sorption of water vapor in dehydrated special foods, NLABS

R.H. %	Beef sandwich			Chicken sandwich			Cheese sandwich			Chicken bites			Melba Toast		
	$-\Delta H$	$-\Delta S$	$-\Delta G_{20^\circ C}$	$-\Delta H$	$-\Delta S$	$-\Delta G_{20^\circ C}$	$-\Delta H$	$-\Delta S$	$-\Delta G_{20^\circ C}$	$-\Delta H$	$-\Delta S$	$-\Delta G_{20^\circ C}$	$-\Delta H$	$-\Delta S$	$-\Delta G_{20^\circ C}$
10	308	0.49	164	155	4.61	200	1110	3.20	120	1060	2.95	196	811	2.27	145
20	439	0.77	223	1920	5.64	267	1510	4.49	192	1420	3.91	274	984	2.70	192
40	582	0.91	315	2140	6.04	370	1940	5.57	304	1790	4.81	381	1170	3.11	258
60	667	0.85	418	2400	6.56	478	2180	6.15	479	1870	4.66	505	1180	2.85	344
80	680	0.35	582	2430	6.14	631	2310	5.90	580	1870	3.95	713	1180	2.44	464
100	680	-0.90	943	2430	5.28	883	2310	4.46	1004	1870	2.80	1050	1180	1.56	721

- ΔH = cal/100g N.F.D.
- ΔS = cal/100g N.F.D./deg
- ΔG = cal/100g N.F.D.

Table 34. Partial molal thermodynamic values for water vapor sorption, and values for relative instrument force of penetration, in pre-cooked freeze-dried beef, S.I.K. II.

R.H. %	$-\overline{\Delta H}$ cal/mole	$-\overline{\Delta G}$ cal/mole	$-\overline{\Delta S}$ cal/mole/degree	Relative force KR for % penetration depth		
				30	63	69
0	7720	-	-	637	855	1250
5	6660	1743	16.8	637	893	1275
12	5850	1285	15.6	677	936	1296
23	4850	855	13.6	638	963	1362
59	970	403	1.94	678	1048	1433
66	0	260	-0.89	720	1130	1451

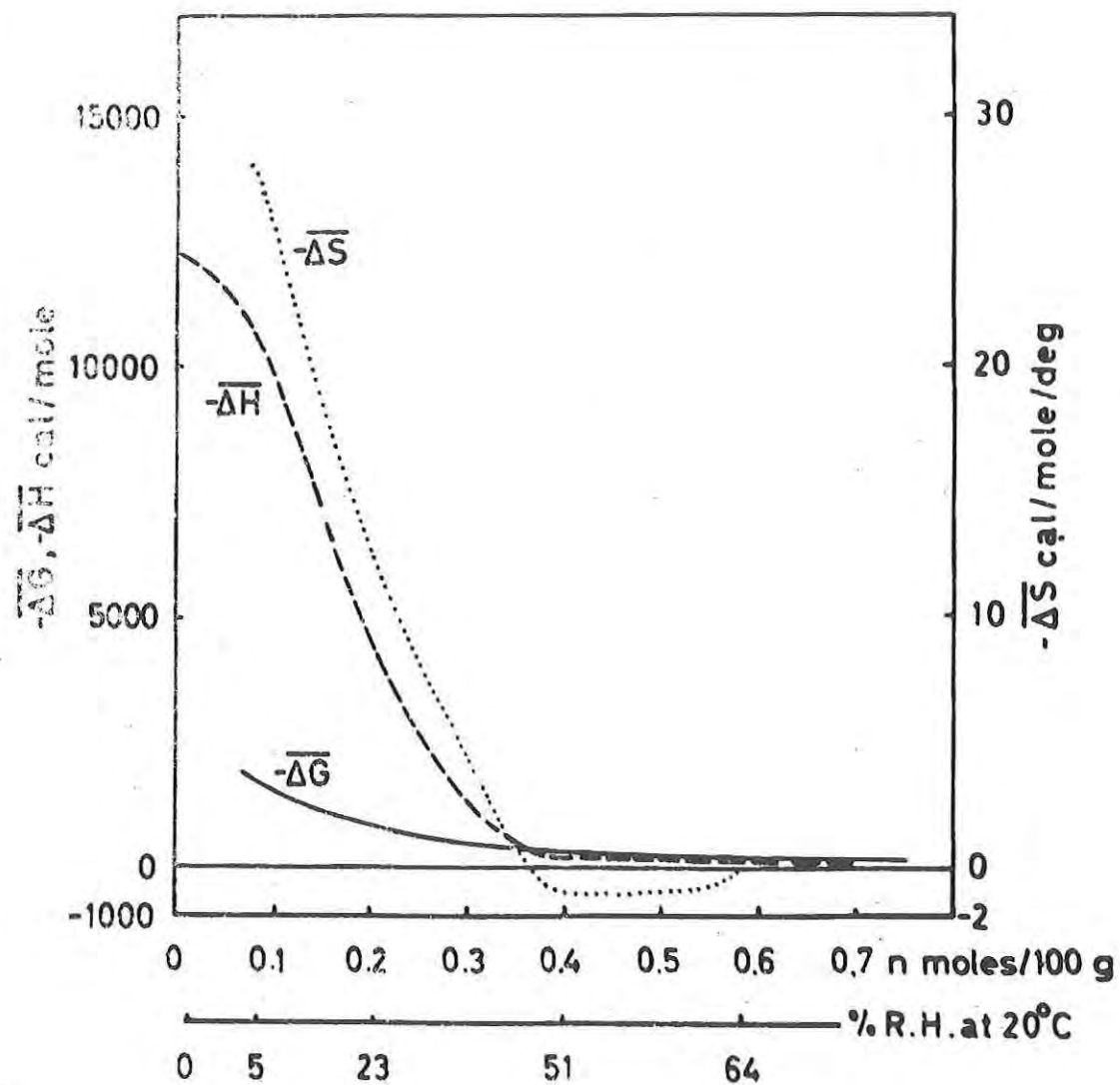


Fig. 37. Curves for partial molal thermodynamic data of water vapor sorption in pre-cooked freeze-dried beef, S.I.K. I.

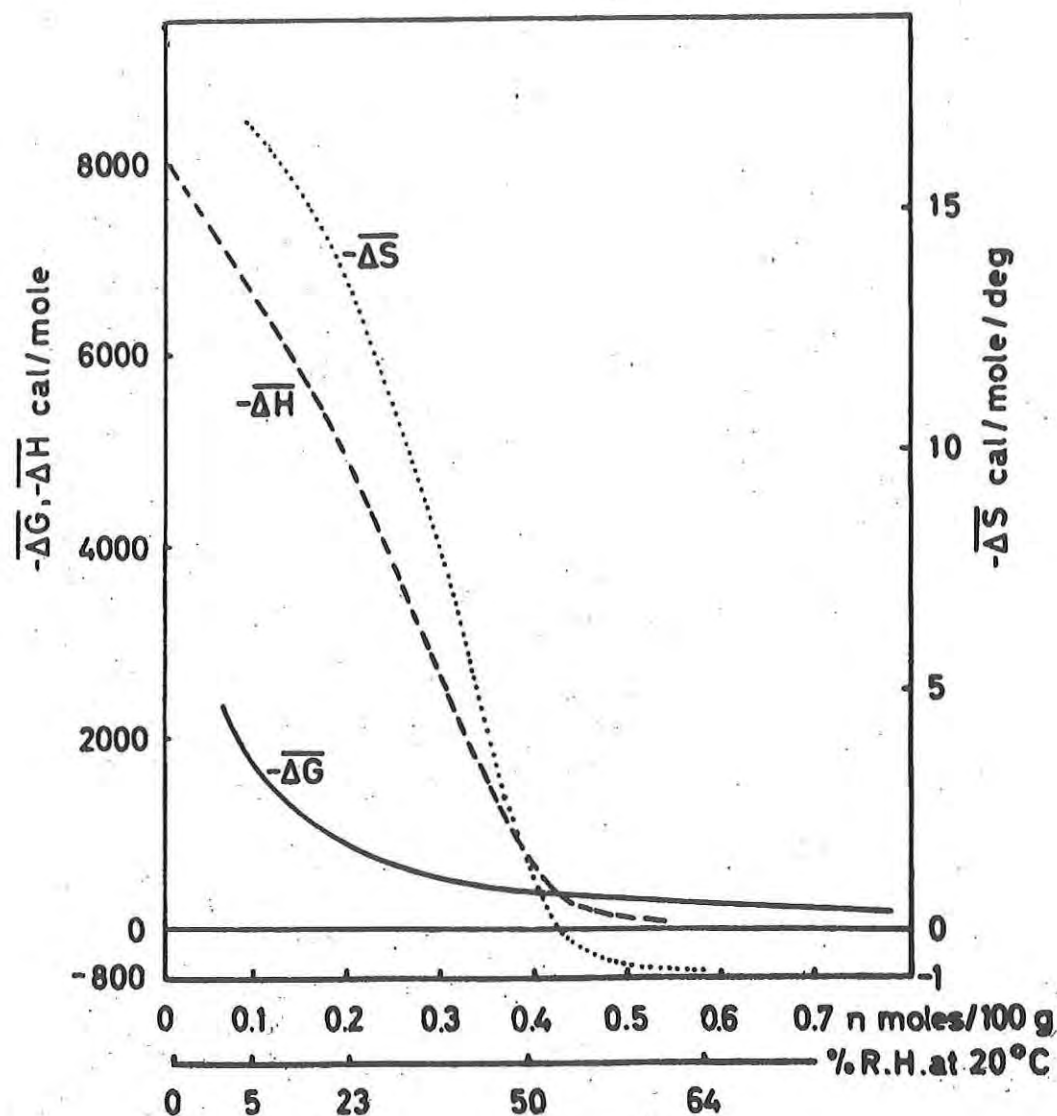


Fig. 38. Curves for partial molal thermodynamic data of water vapor sorption in pre-cooked freeze-dried beef, S.I.K. II.

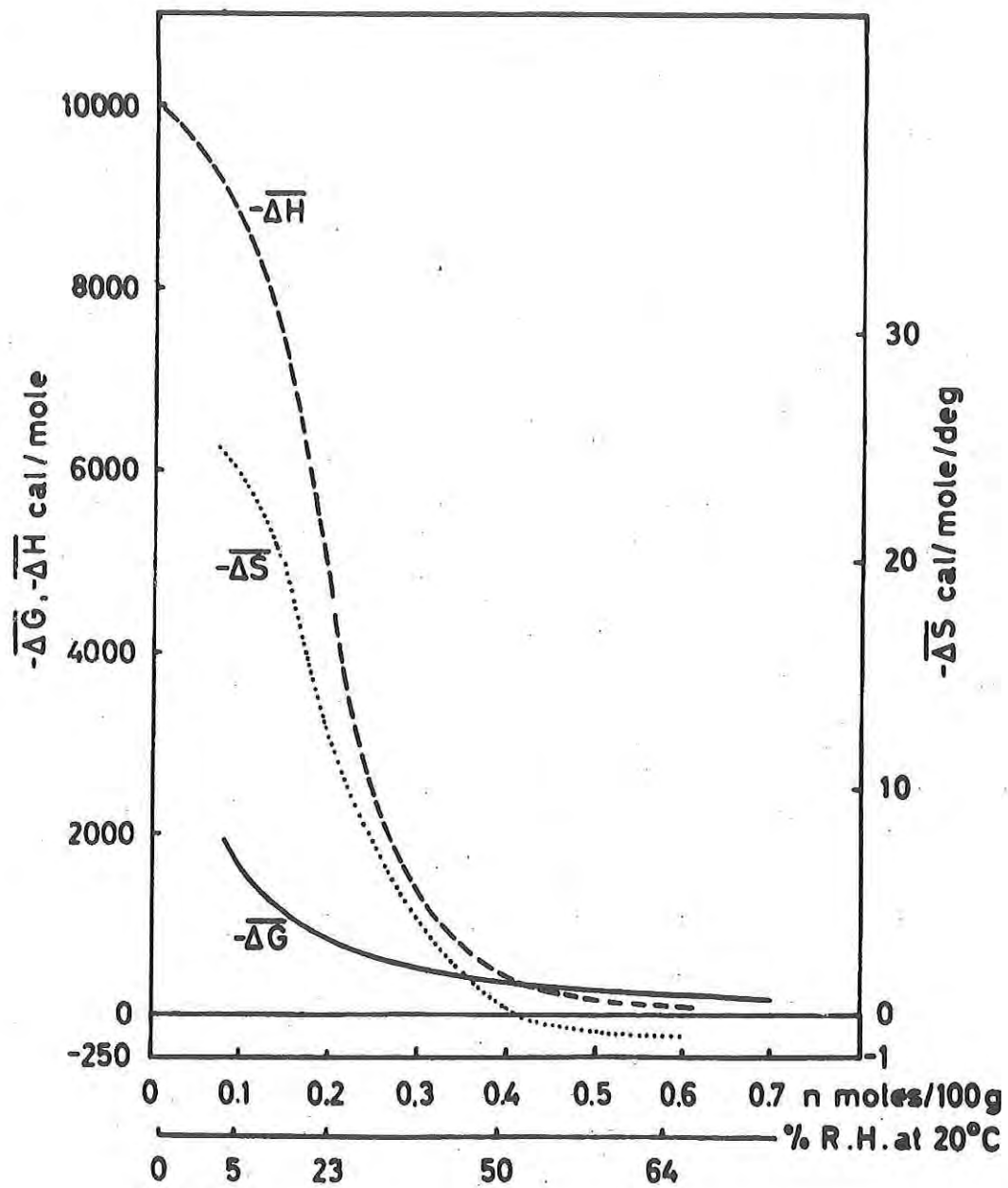


Fig. 39. Curves for partial molal thermodynamic data of water vapor sorption in pre-cooked freeze-dried beef, S.I.K. III.

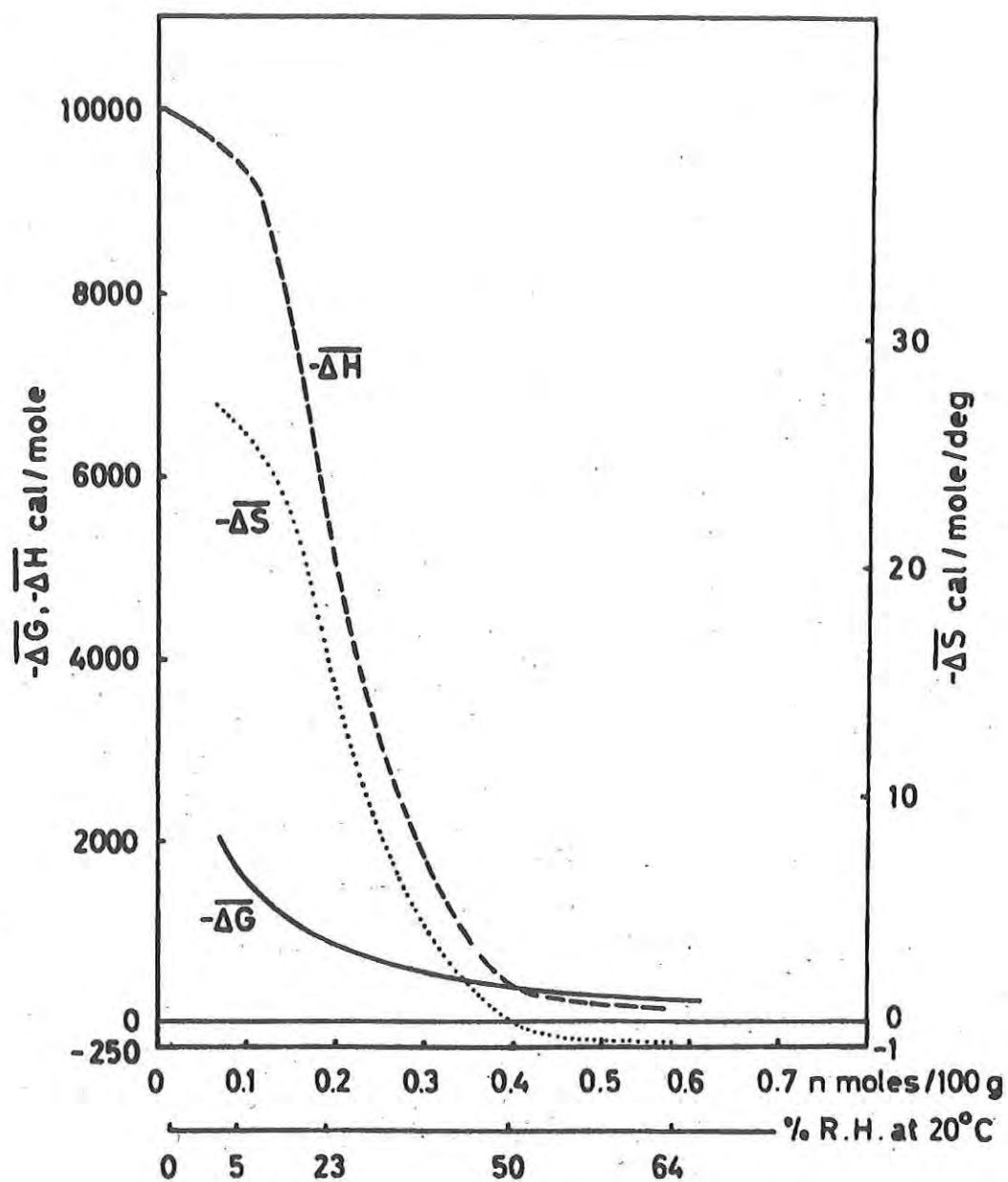


Fig. 40. Curves for partial molal thermodynamic data of water vapor sorption in pre-cooked freeze-dried beef, S.I.K. IV.

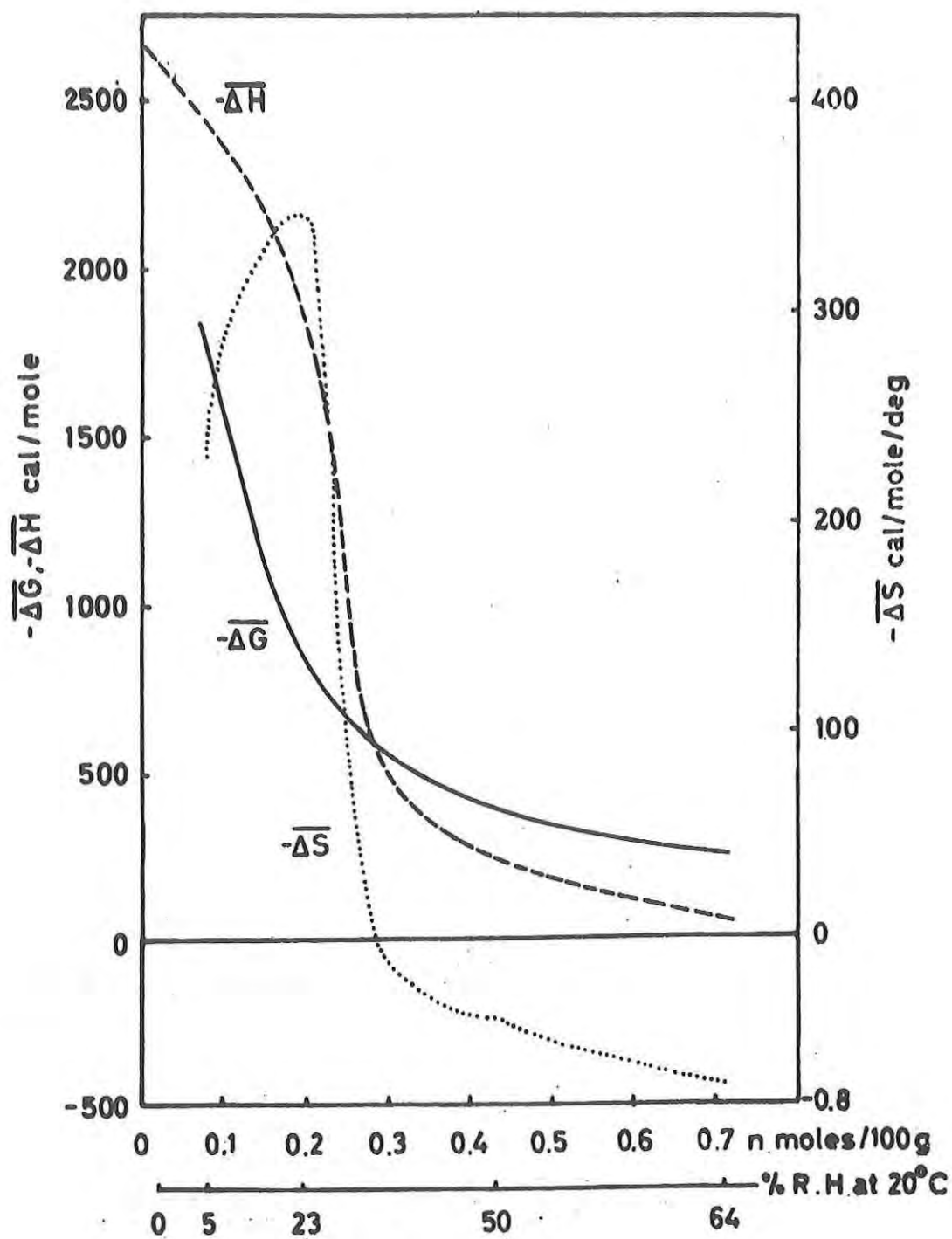


Fig. 41. Curves for partial molal thermodynamic data of water vapor sorption in dehydrated beef sandwich.

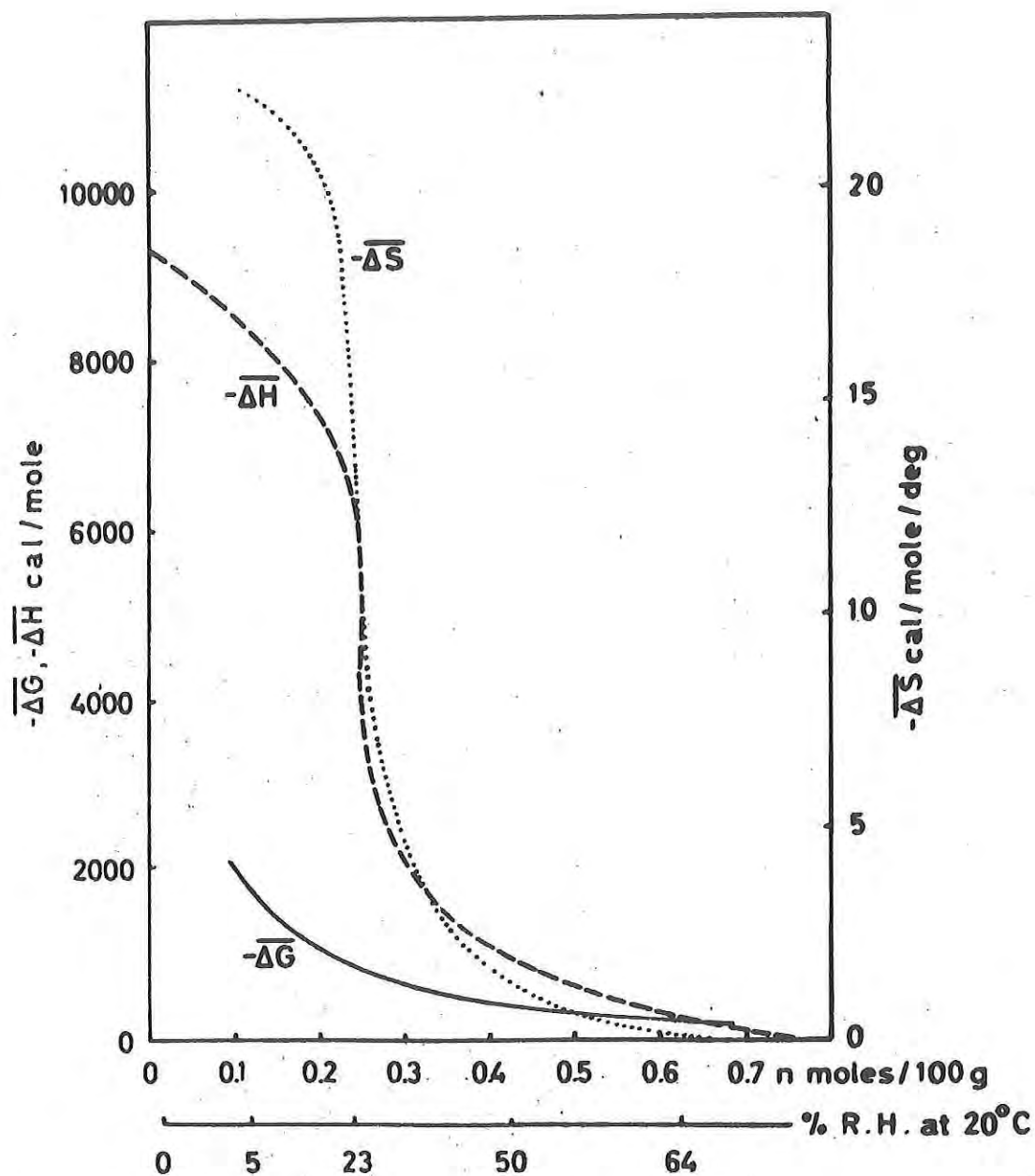


Fig. 42. Curves for partial molal thermodynamic data of water vapor sorption in dehydrated chicken sandwich.

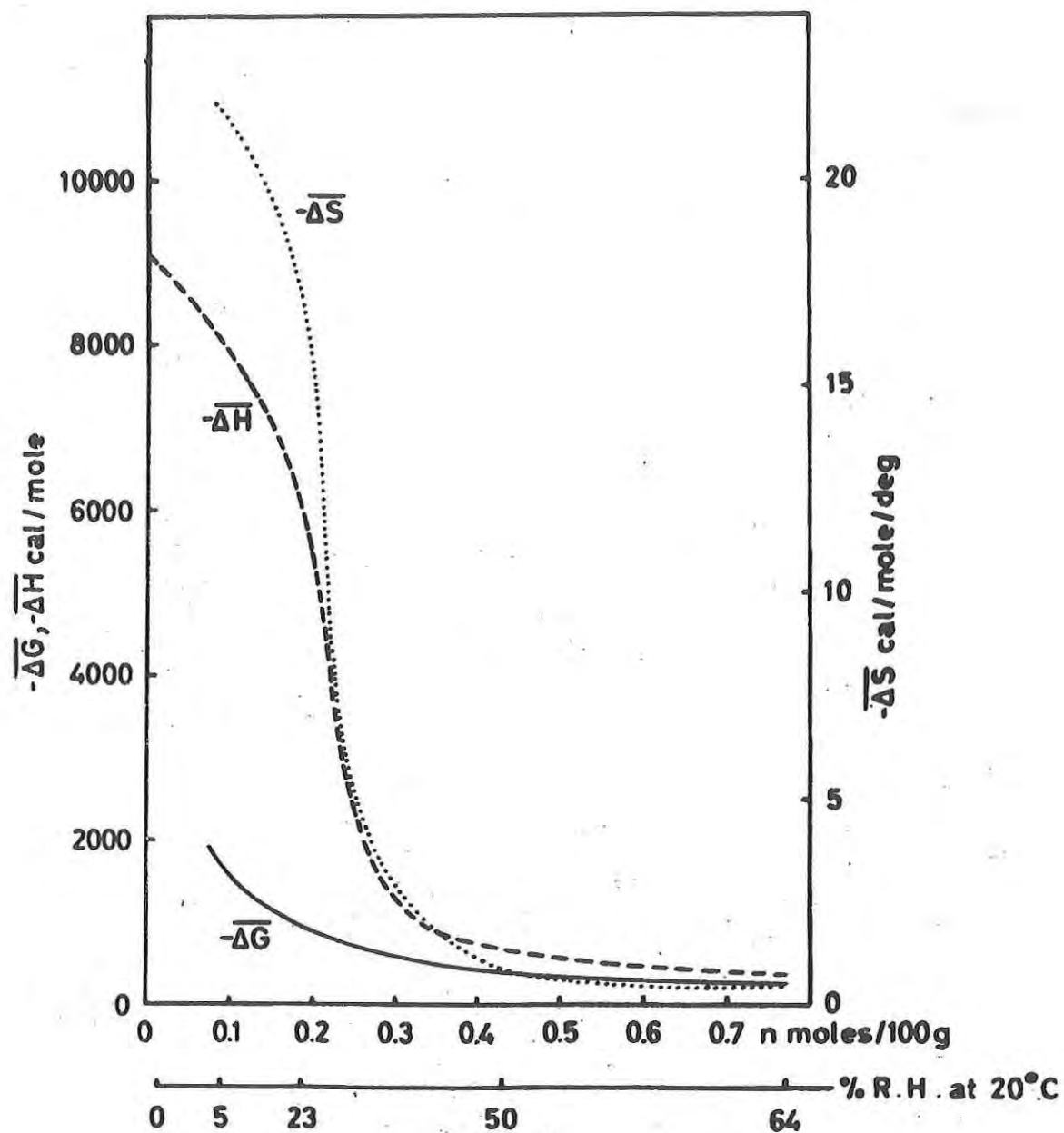


Fig. 43. Curves for partial molal thermodynamic data of water vapor sorption in dehydrated cheese sandwich.

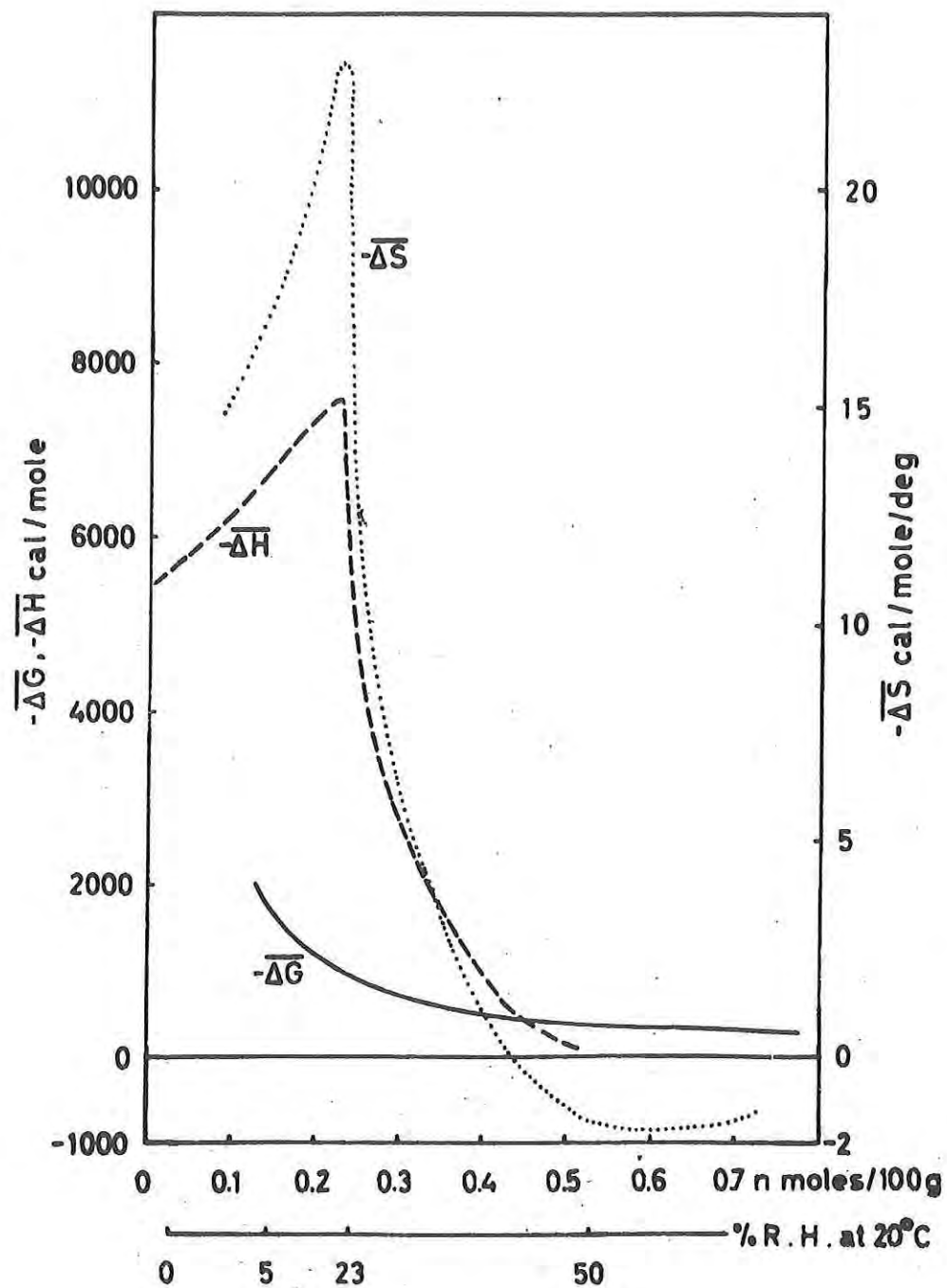


Fig. 44. Curves for partial molal thermodynamic data of water vapor sorption in dehydrated chicken bites.

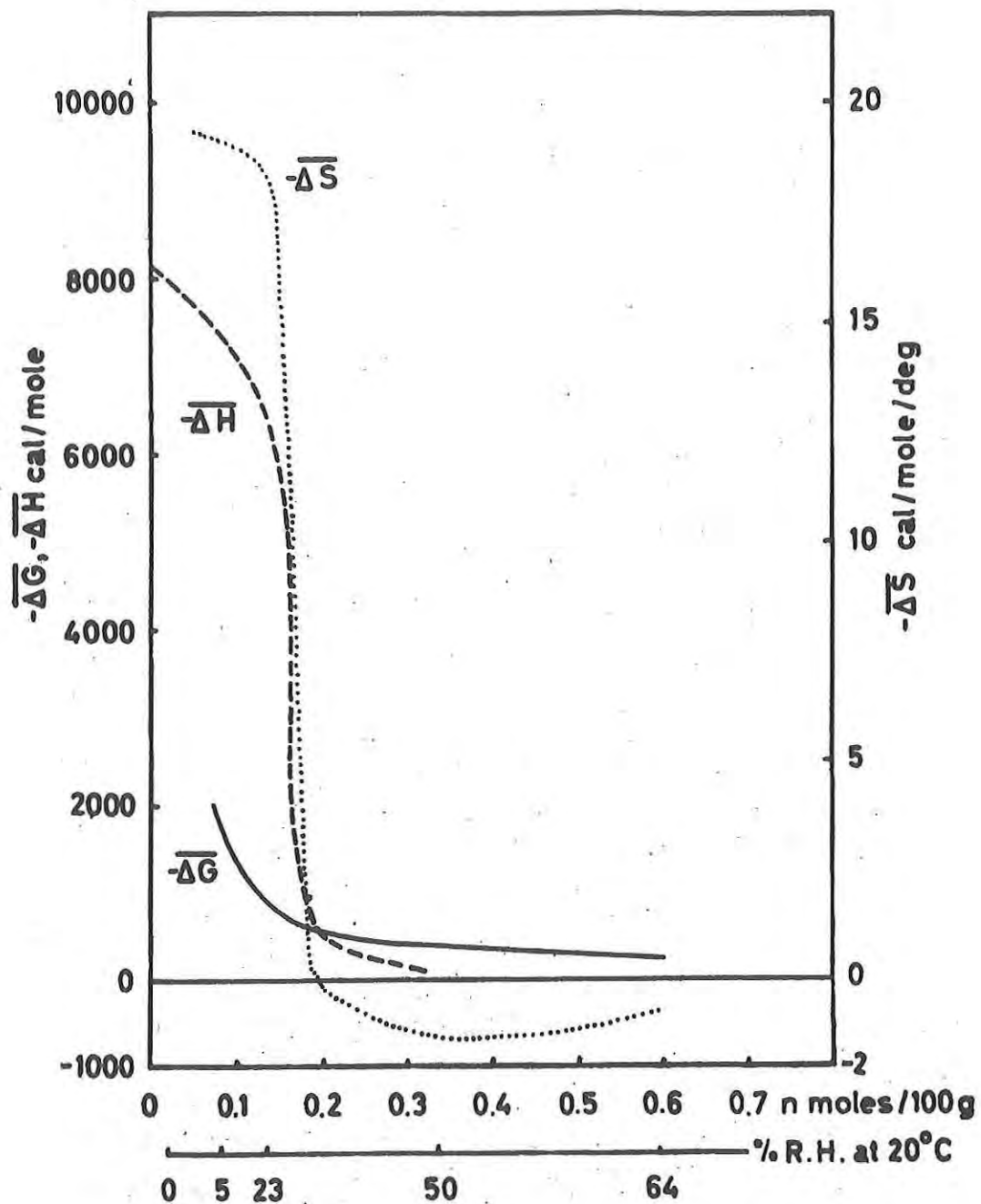
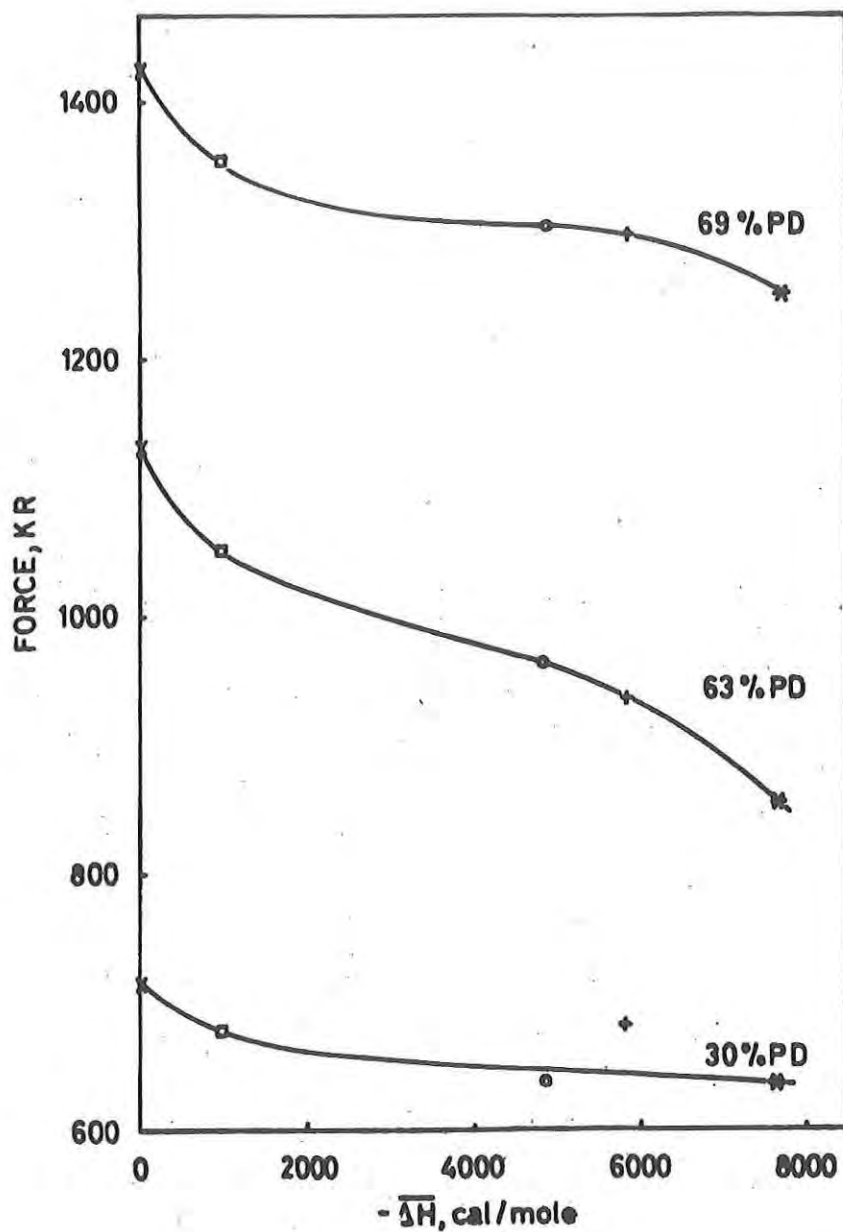
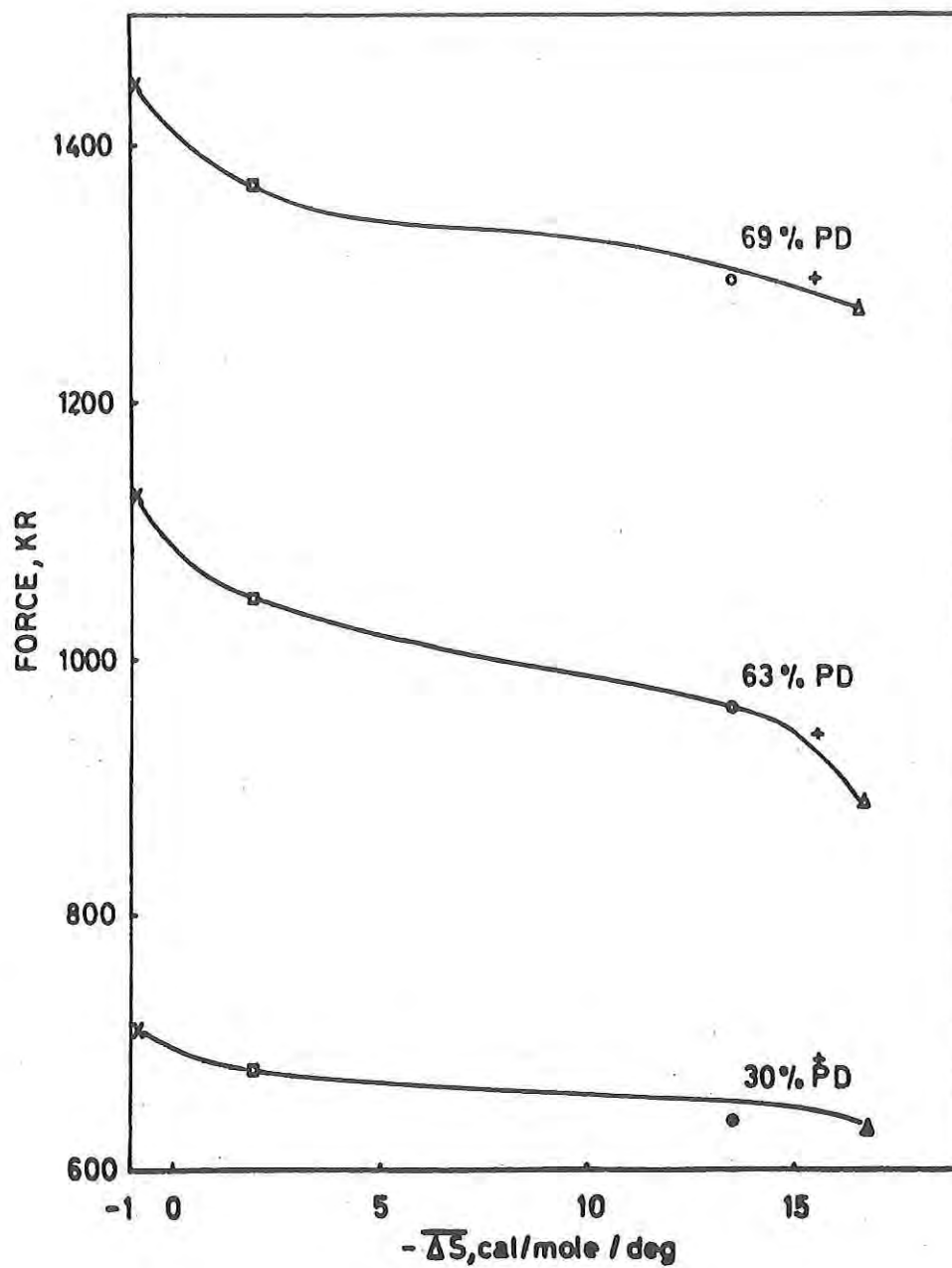


Fig. 45. Curves for partial molal thermodynamic data of water vapor sorption in melba toast.



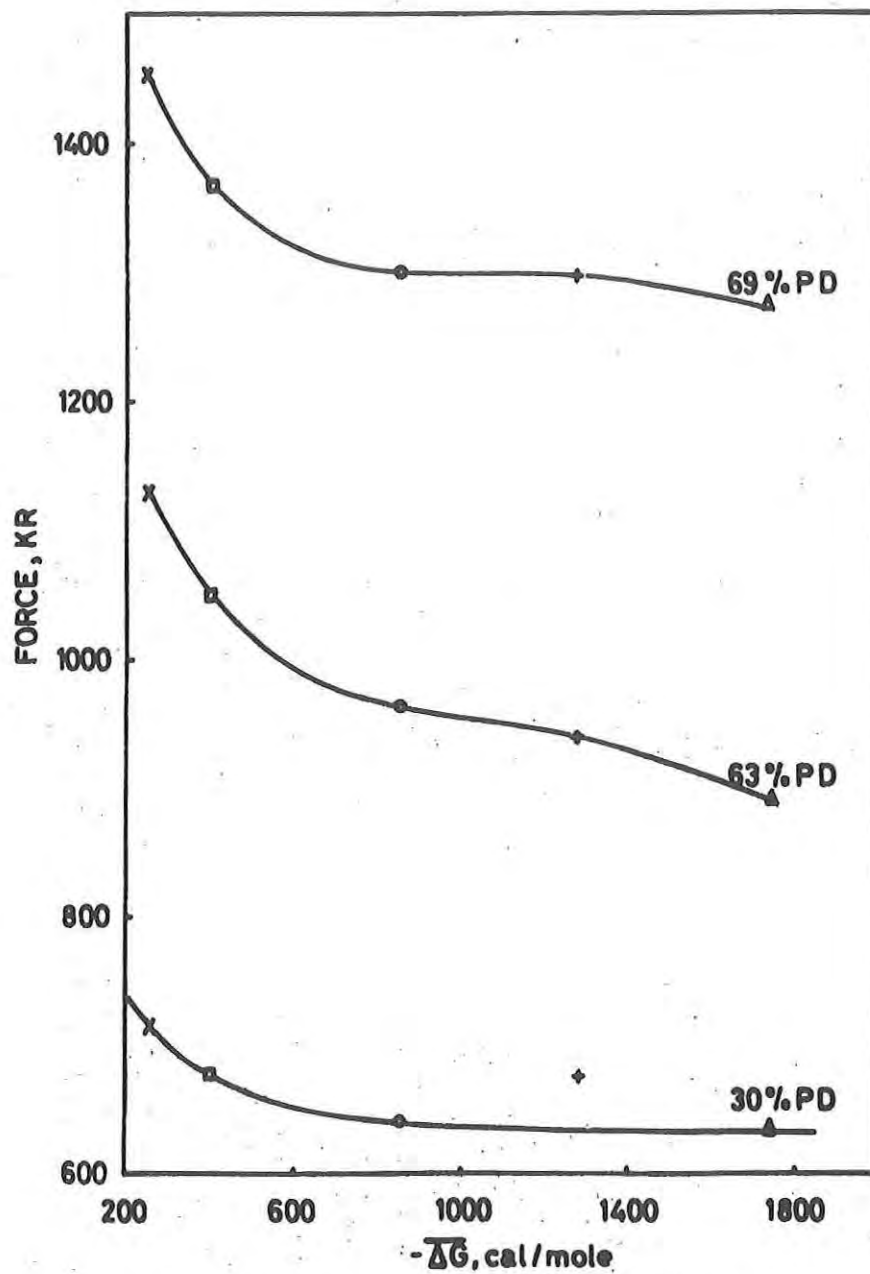
* = 0, + = 12, o = 23, □ = 50, x = 66 % R.H. at 20°C

Fig. 46. Plot of values for the partial molal enthalpy change of water vapor sorption vs. values for instrument force of penetration in pre-cooked freeze-dried beef, S.I.K. II.



$\Delta = 5, + = 12, o = 23, \square = 50, x = 66$ % R.H. at 20°C

Fig. 47. Plot of values for the partial molal entropy change of water vapor sorption vs. values for instrument force of penetration in pre-cooked freeze-dried beef, S.I.K. II.



△ = 5, ◇ = 12, ○ = 23, □ = 50, x = 66 % R.H. at 20°C

Fig. 48. Plot of values for the partial molal free energy change of water vapor sorption vs. values for instrument force of penetration in pre-cooked freeze-dried beef, S.I.K. II.

RAW FREEZE-DRIED BEEF PROCESSED BY S.I.K.

Figs. 49 and 50 show the relationships between elastic, crushing and total texture behavior vs. the penetration depth in two different samples A and B of raw freeze-dried beef, in the dry and rehydrated states. Figs. 51 and 52 show the same parameters for the rehydrated samples A and B on a larger scale, and Fig. 53 for a different sample C in the rehydrated state.

In general, the differences within the dry and rehydrated samples are quantitative rather than qualitative. In the dry samples, if the curves are extrapolated to zero PD, where each parameter is zero, it becomes evident that they are more or less of a sigmoid shape. It is obvious that the selection of the depth of penetration can be important in the objective measurement of textural characteristics, especially with respect to the total and crushing texture behavior. In the rehydrated samples the curves are generally convex to both axes. At high penetration depths the value of all textural parameters increases sharply with depth. As discussed previously in the case of pre-cooked freeze-dried meat, this is due to the pressing or compacting of the dry meat under the punch.

As expected, all of the above mentioned parameters are smaller in the rehydrated than in the dry food, the value Σ showing this difference most dramatically. In Fig. 49, for example, the value E at about 20% PD of the dry material is about equal to the value Σ at 75% PD of the rehydrated food. In both the dry and the reconstituted product the value E , which is a function of the elastic work, is smaller than the corresponding value Δ , which is a function of the irreversible crushing work.

Fig. 54 shows the relationship between hardness and penetration depth of the above samples A, B and C in the dry and rehydrated states. Fig. 55 presents the results on the rehydrated meat on a larger scale. The dry samples B and C behaved similarly, with hardness showing only a little change between about 25 and 55 % PD. Within this range, any PD could be selected for experi-

mental measurements. Above about 55 % PD, the hardness progressively increased due to the above mentioned compacting of the meat under the punch. The dry sample A behaved somewhat differently, with hardness increasing continuously from about 20 to 65 % PD. In this case, it is important at what penetration depth hardness is measured. Dry sample A was consistently harder than dry samples B and C. In the rehydrated state (Fig. 55), sample A became softer than sample C and generally harder than sample B. Since these slices were derived from the same muscle of the same animal, although not necessarily from adjacent positions within the muscle, the results indicate the importance of representative sampling and of statistical treatment of data, in any quality control or grading program for the texture of raw meat.

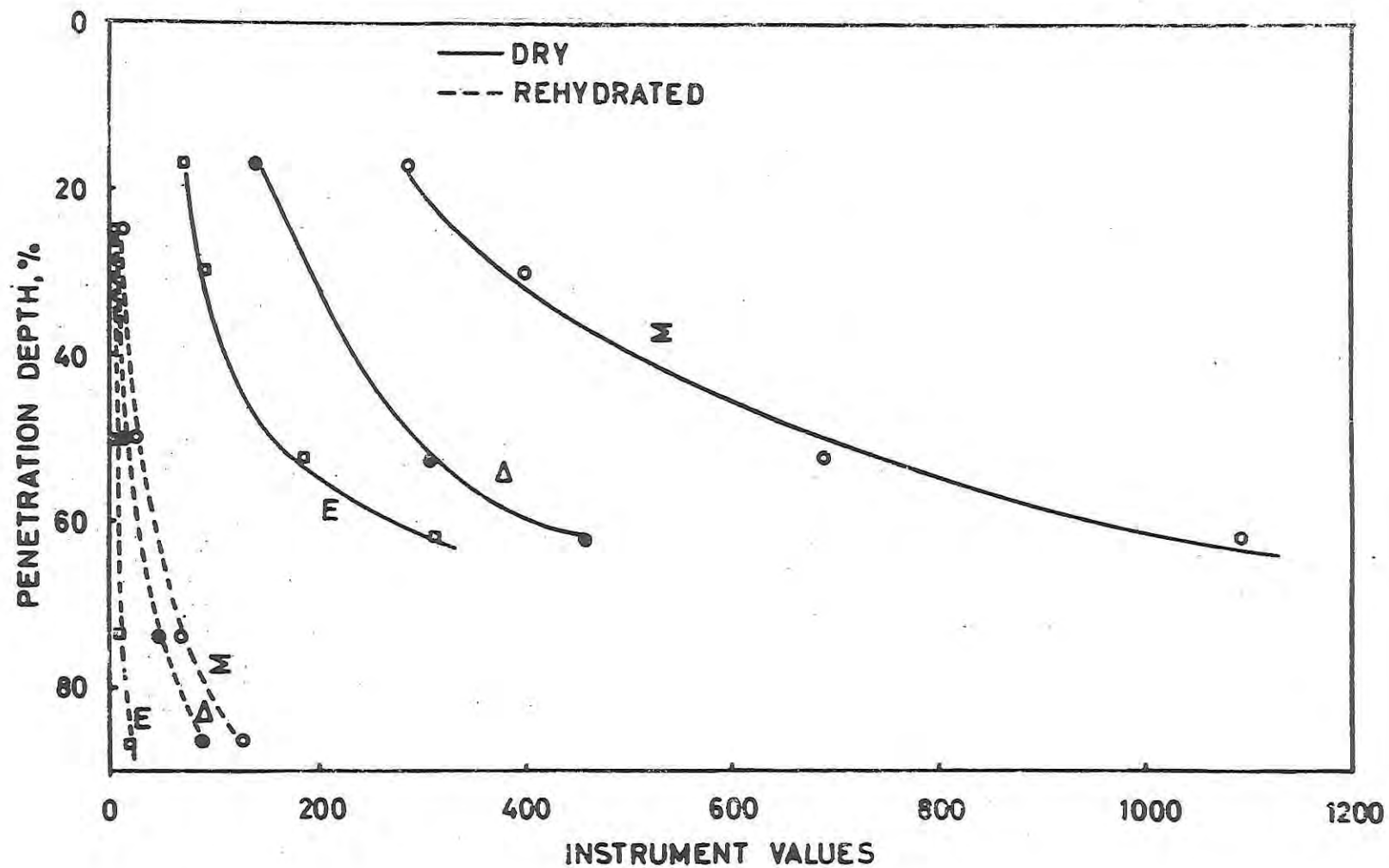


Fig.49. Total Σ , Crushing Δ and Elastic E texture behavior of raw freeze-dried beef, in the dry and rehydrated states.

Sample A.

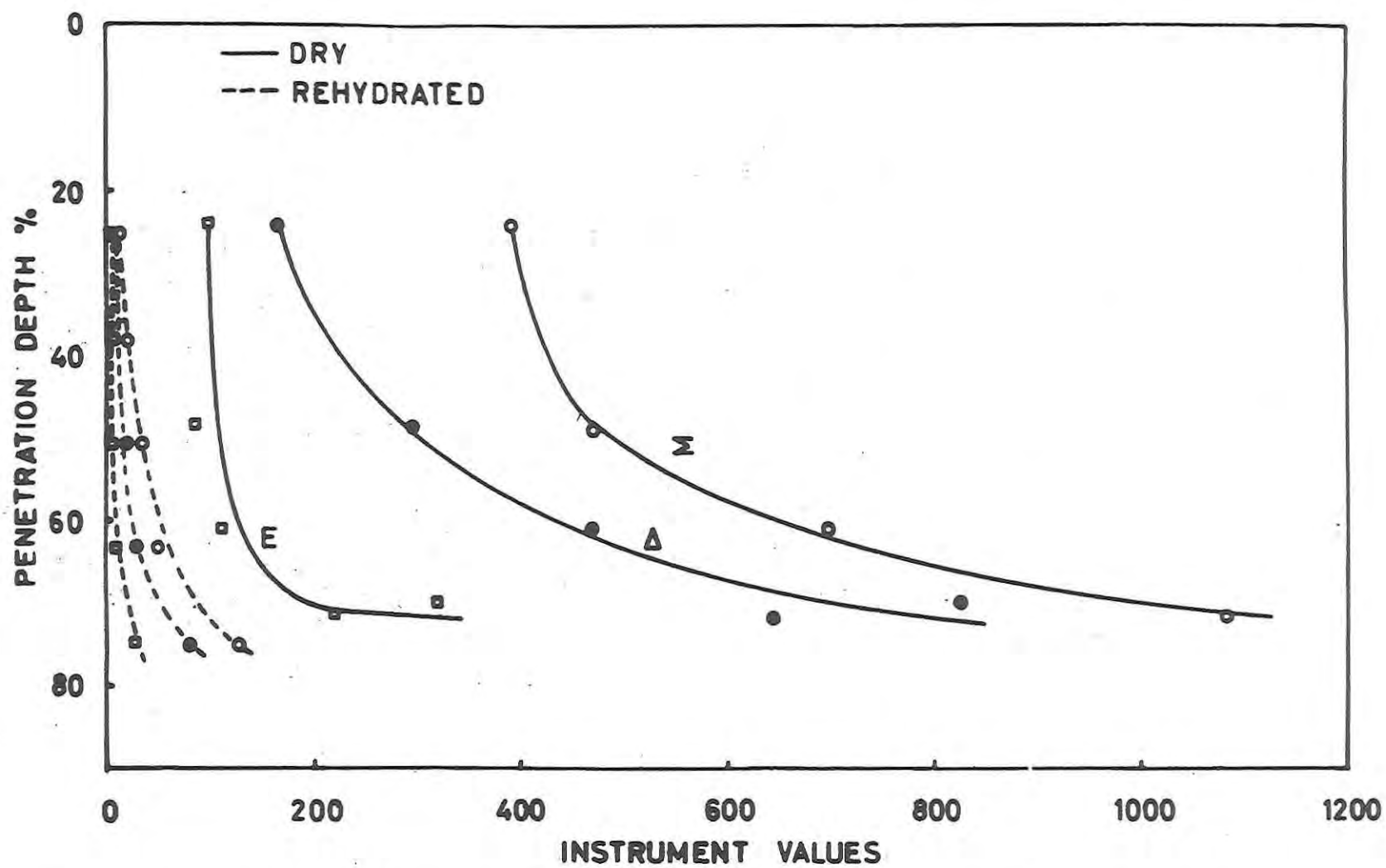


Fig.50. Total Σ , Crushing Δ and Elastic E texture behavior of raw freeze-dried beef, in the dry and rehydrated states.

Sample B.

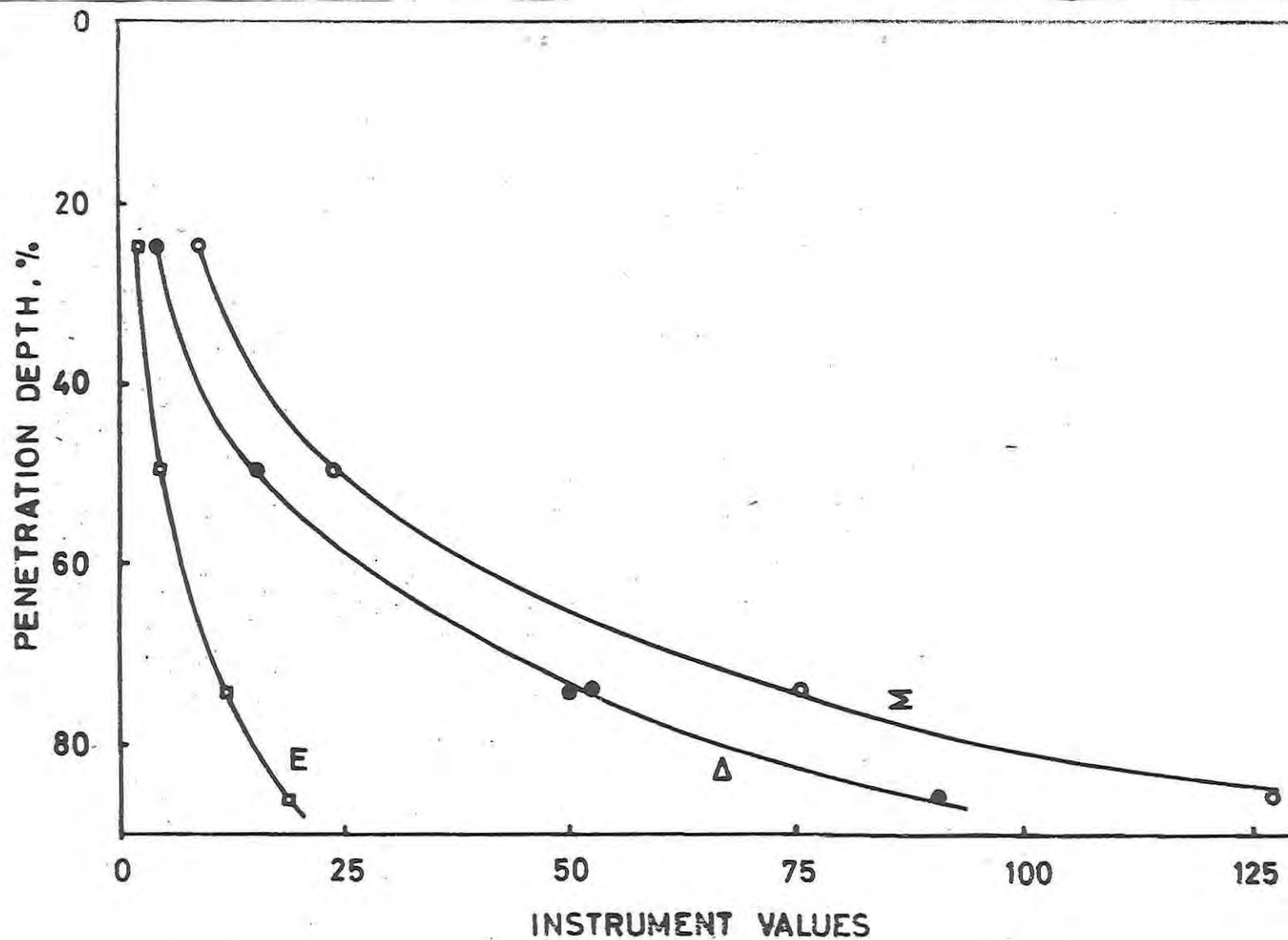


Fig. 51. Total Σ , Crushing Δ and Elastic E texture behavior of raw freeze-dried beef, in the rehydrated state.

Sample A.

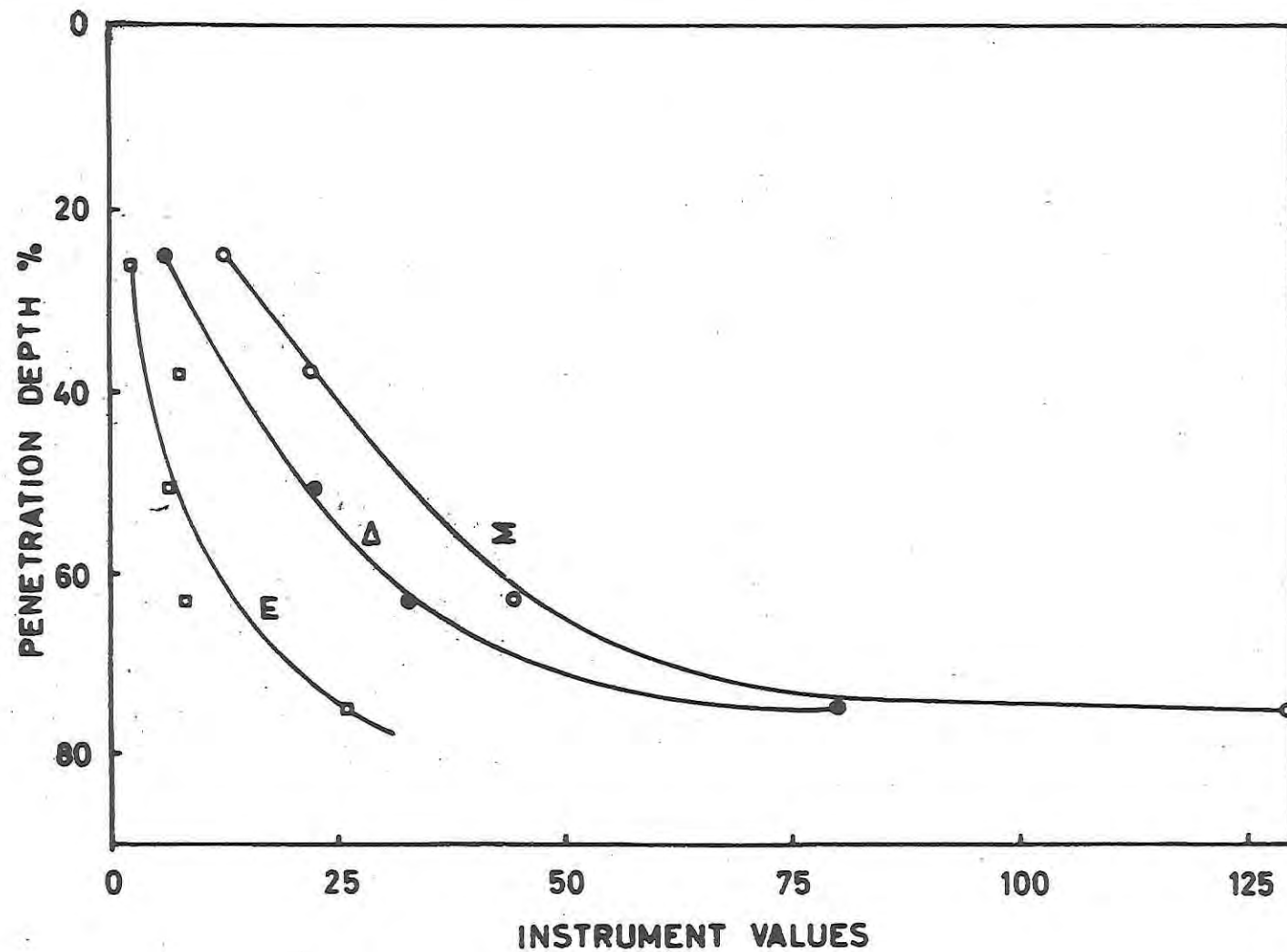


Fig. 52. Total Σ , Crushing Δ and Elastic E texture behavior of raw freeze-dried beef, in the rehydrated state.

Sample B.

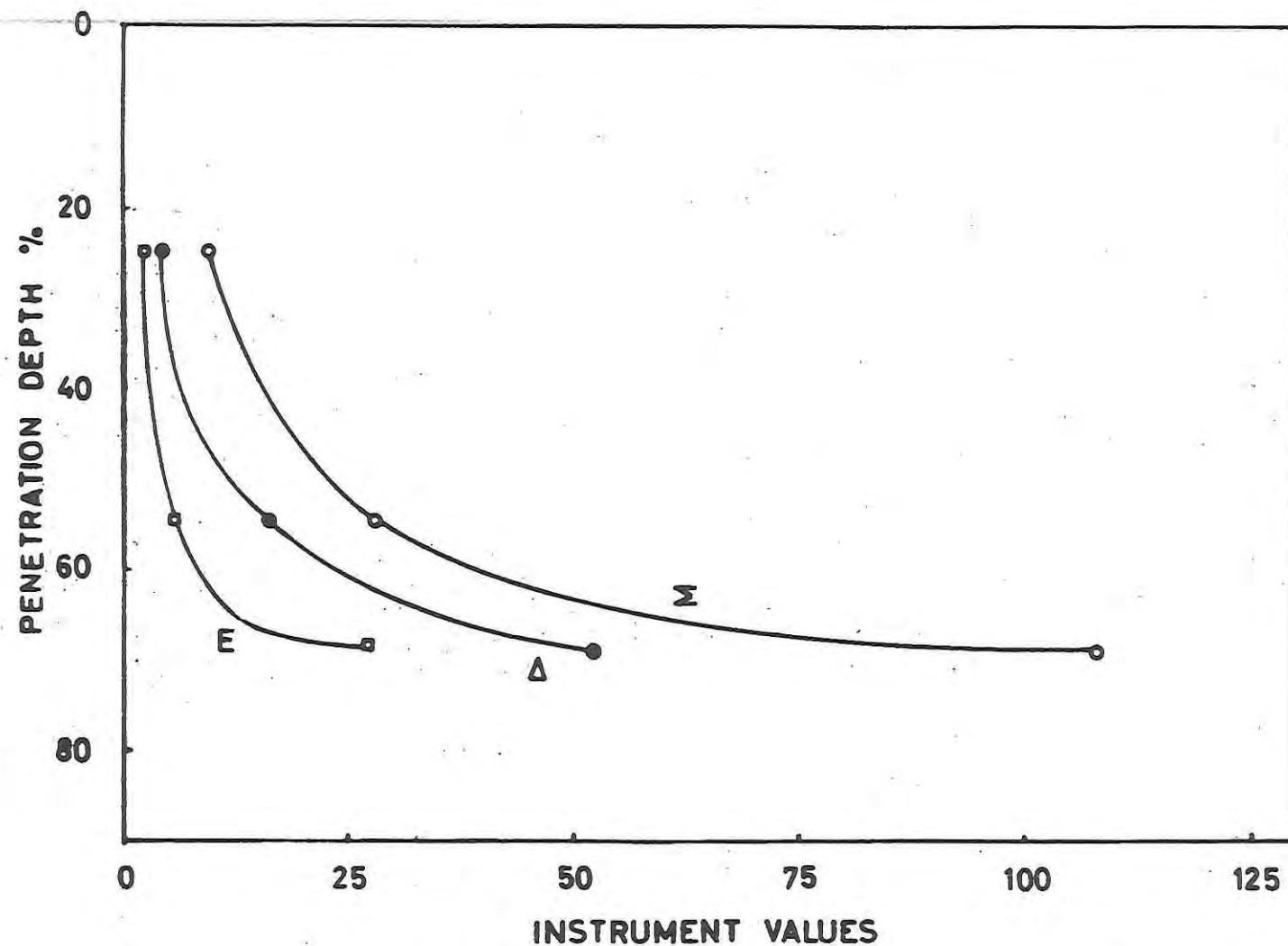


Fig. 53. Total Σ , Crushing Δ and Elastic E texture behavior of raw freeze-dried beef, in the rehydrated state.

Sample C.

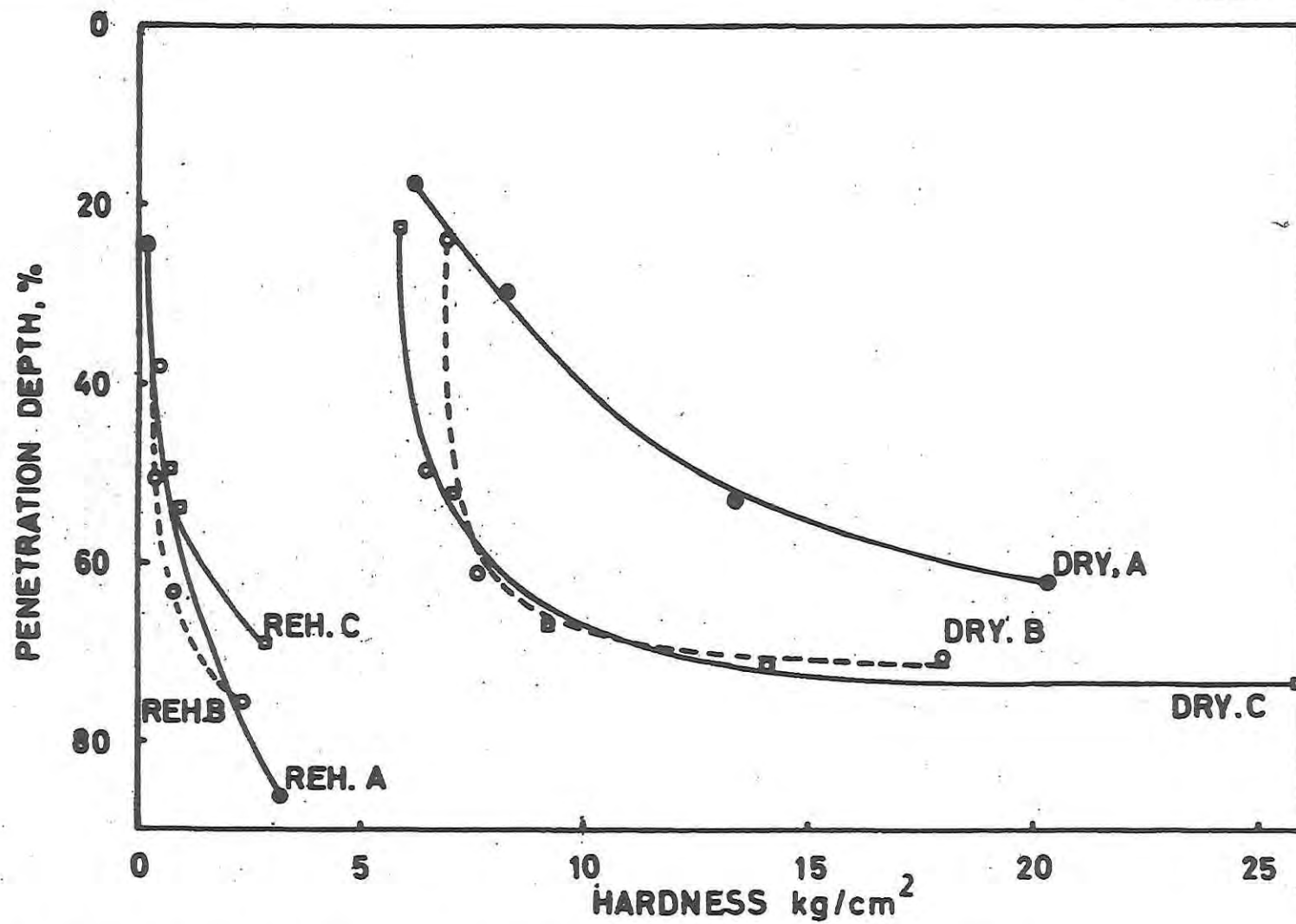


Fig. 54. Hardness of raw freeze-dried beef in the dry and rehydrated states.

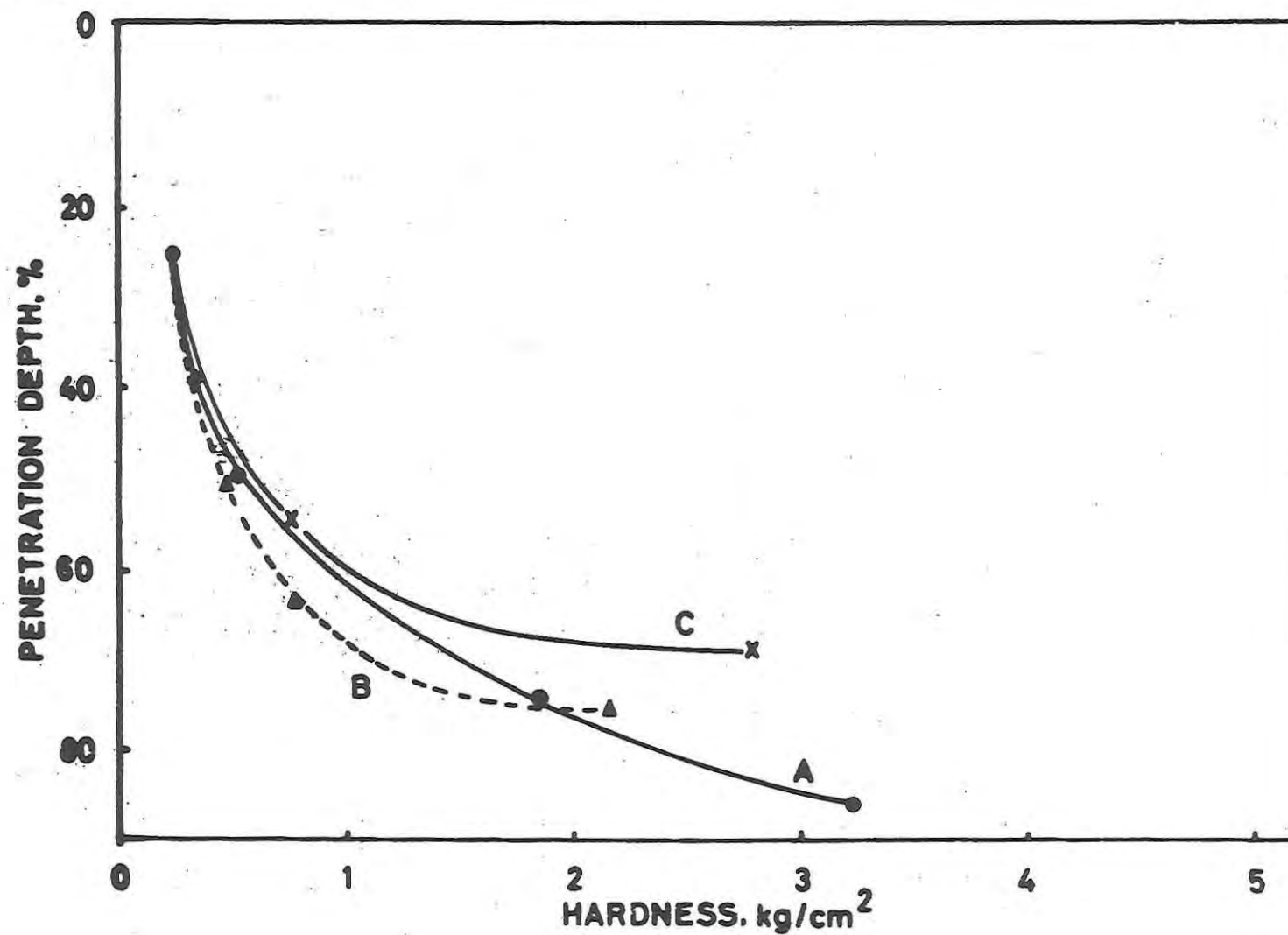


Fig. 55. Hardness of raw freeze-dried beef in the rehydrated state.

SENSORY EVALUATION

Table 35 and Figs. 56-58 show the relationship between relative humidity and ratings for the different sensory parameters. The results are in close agreement with those obtained for the relationship between relative humidity and mechanical texture parameters. In general, the higher the relative humidity the tougher the meat, in both the dry and the rehydrated state.

Results on the relationship between the partial molal enthalpy change of water vapor sorption and sensory texture parameters for the S.I.K. III sample series are plotted in Figs. 59-63. In general, the ratings for the different sensory texture parameters are in close agreement with the Mastico-meter values for the mechanical parameters. Lower partial molal enthalpy changes, (which correspond to higher relative humidity and moisture values), are associated with more tough meat, both before and after rehydration in the mouth. The reasons for the increase in toughness with increasing hygroscopic equilibrium values have been discussed previously.

An interesting aspect of the sensory evaluation is the close relationship between the heat evolved in the mouth during mastication and the integral net enthalpy change, as shown in Table 36 and Fig. 64. The reliability of testing by the sensory panel, and the close control of the experimental variables in the work of instrumental measurements will be shown further in the chapter on the statistical analysis of data.

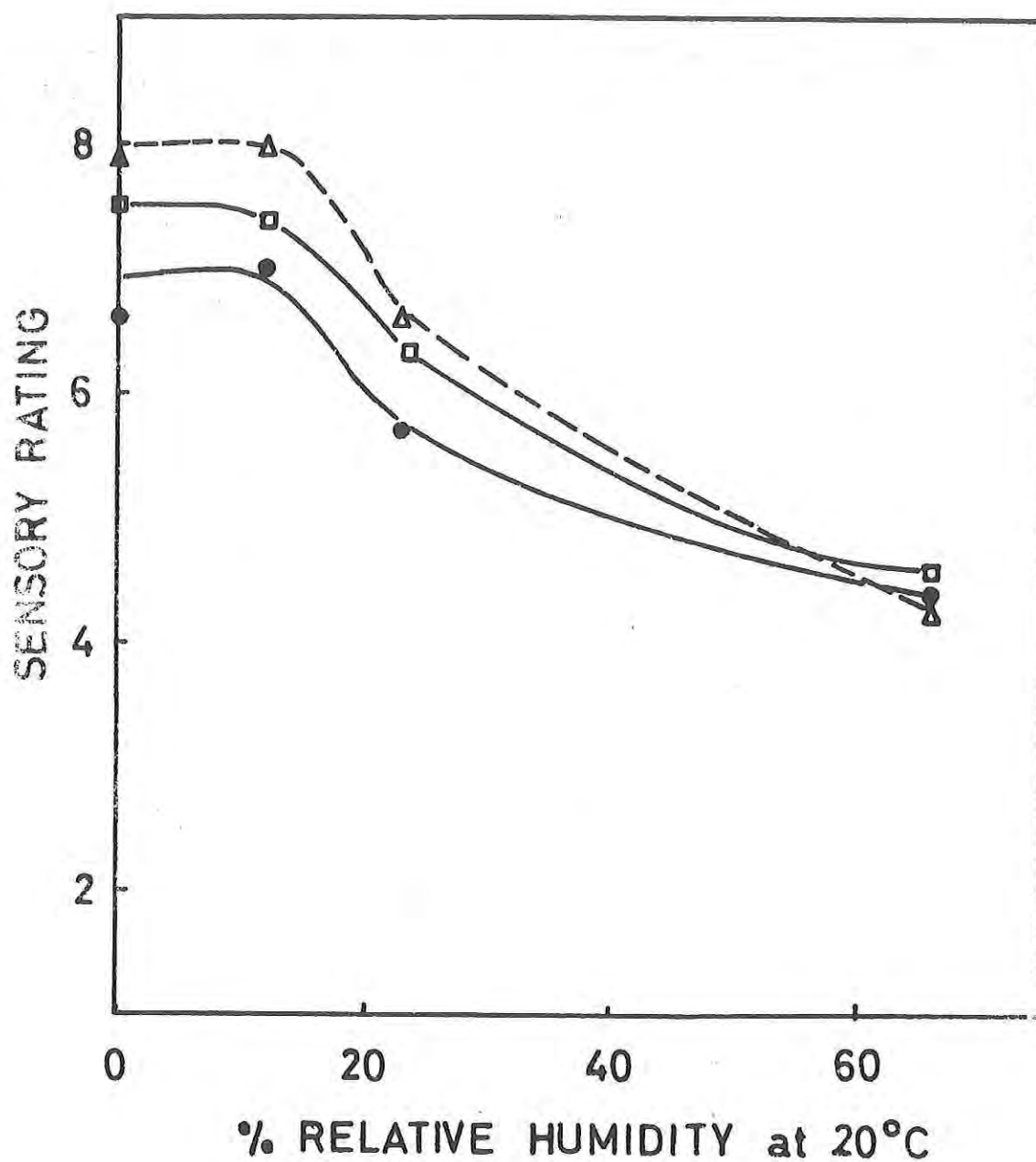
Table 35. Values of sensory ratings of pre-cooked freeze-dried beef, S.I.K. II and IV.

% relative humidity at 20°C	Hardness before rehydration in mouth				Tenderness after rehydration in mouth		Flavor after rehydration in mouth		Heat evolved in mouth during mastication	
	parallel to fibers		across fibers		III	IV	III	IV	III	IV
	III	IV	III	IV						
0	7.50	7.81	7.87	8.00	6.56	6.62	5.25	5.06	2.4	2.3
12.	7.43	7.43	8.00	8.12	7.00	6.00	5.06	4.56	1.9	1.8
23	6.31	6.62	6.56	6.93	5.68	6.18	4.87	4.37	1.1	0.9
66	4.56	4.00	4.25	3.93	4.37	4.06	3.31	2.68	0.4	0.3
Mean	6.45	6.46	6.67	6.75	5.90	5.71	4.62	4.17	-	-

Table 36. Sensory ratings for heat evolved in mouth during mastication, and values of the integral net heat of water vapor sorption * in pre-cooked freeze-dried beef, S.I.K. III

R.H. %	$(-\Delta H)^*$ cal/100 g N.F.D.	Sensory rating for heat evolved in mouth
0	1960	2.4
12	770	1.9
23	420	1.1
66	10	0.4

* ΔH at saturation minus ΔH at the corresponding % R.H.



□ = hardness determined parallel to meat fibers before rehydration in mouth.

1 = extremely difficult to chew, 9 = extremely easy to chew.

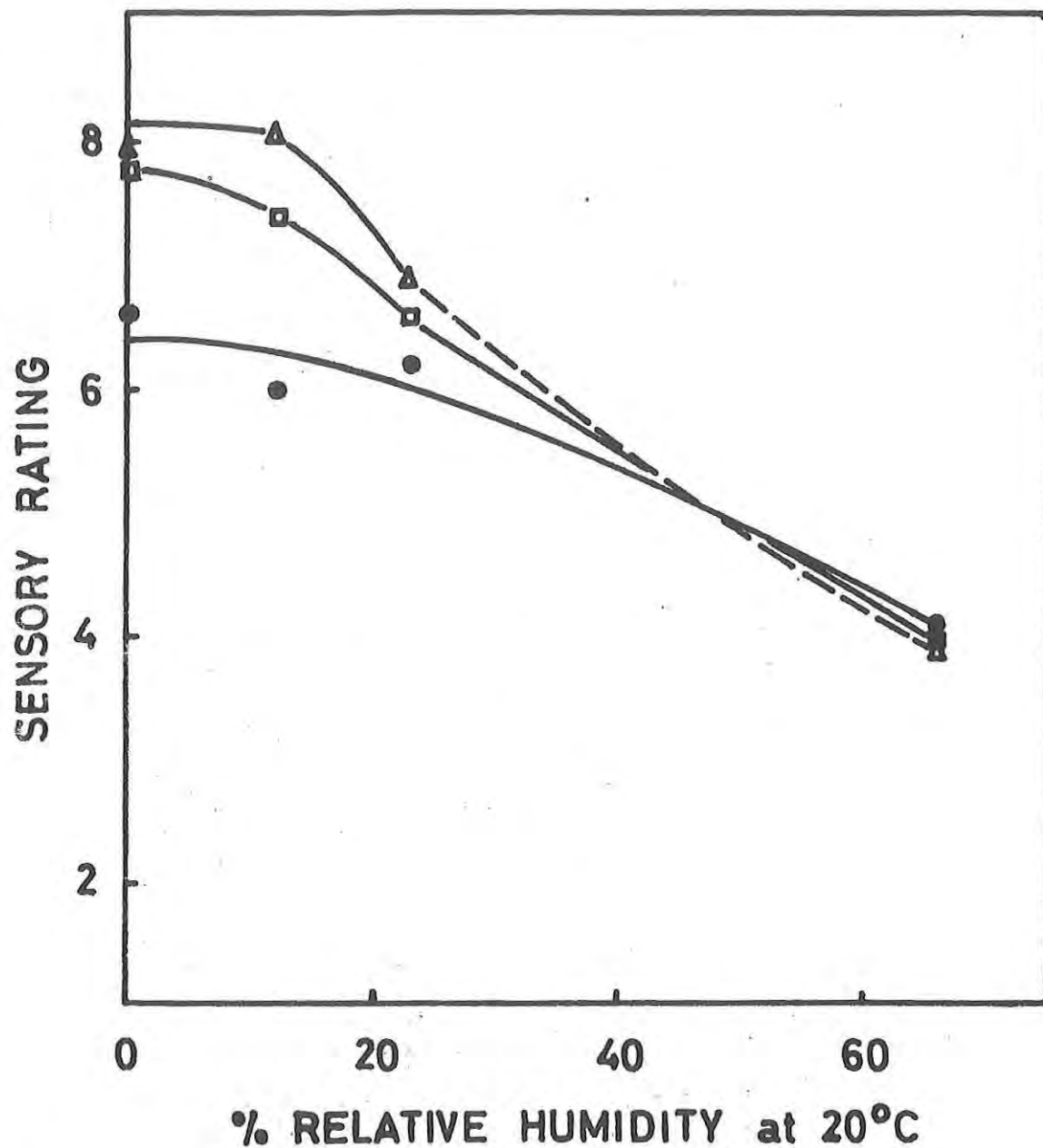
△ = hardness determined across meat fibers before rehydration in mouth.

1 = extremely difficult to chew, 9 = extremely easy to chew.

● = tenderness after rehydration in mouth.

1 = extremely tough, 9 = extremely tender.

Fig. 56. Plot of sensory ratings vs. values of % relative humidity in pre-cooked freeze-dried beef, S.I.K. III.



- = hardness determined parallel to meat fibers before rehydration in mouth.
 1 = extremely difficult to chew, 9 = extremely easy to chew.
- Δ = hardness determined across fibers before rehydration in mouth.
 1 = extremely difficult to chew, 9 = extremely easy to chew.
- = tenderness after rehydration in mouth.
 1 = extremely tough, 9 = extremely tender.

Fig. 57. Plot of sensory ratings vs. values of % relative humidity in pre-cooked freeze-dried beef, S.I.K. IV.

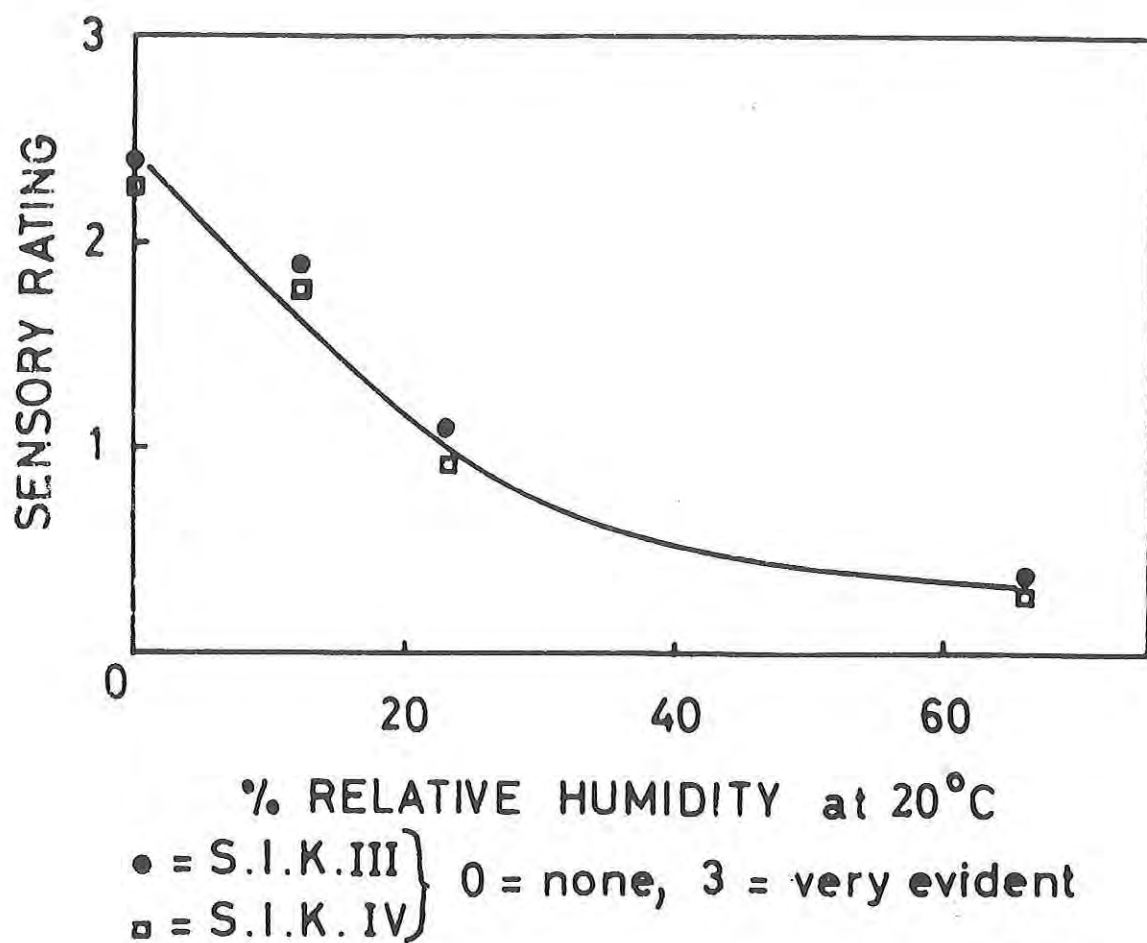
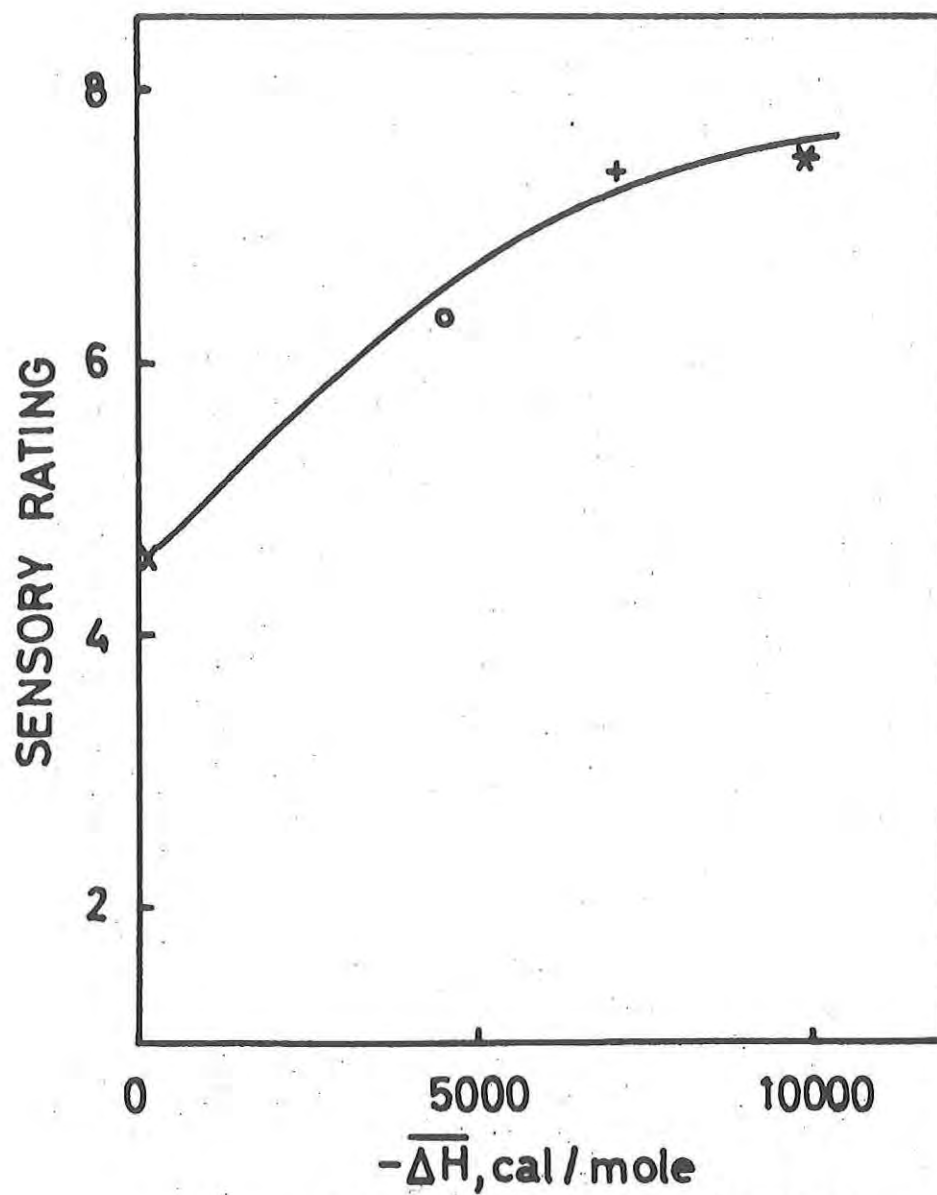
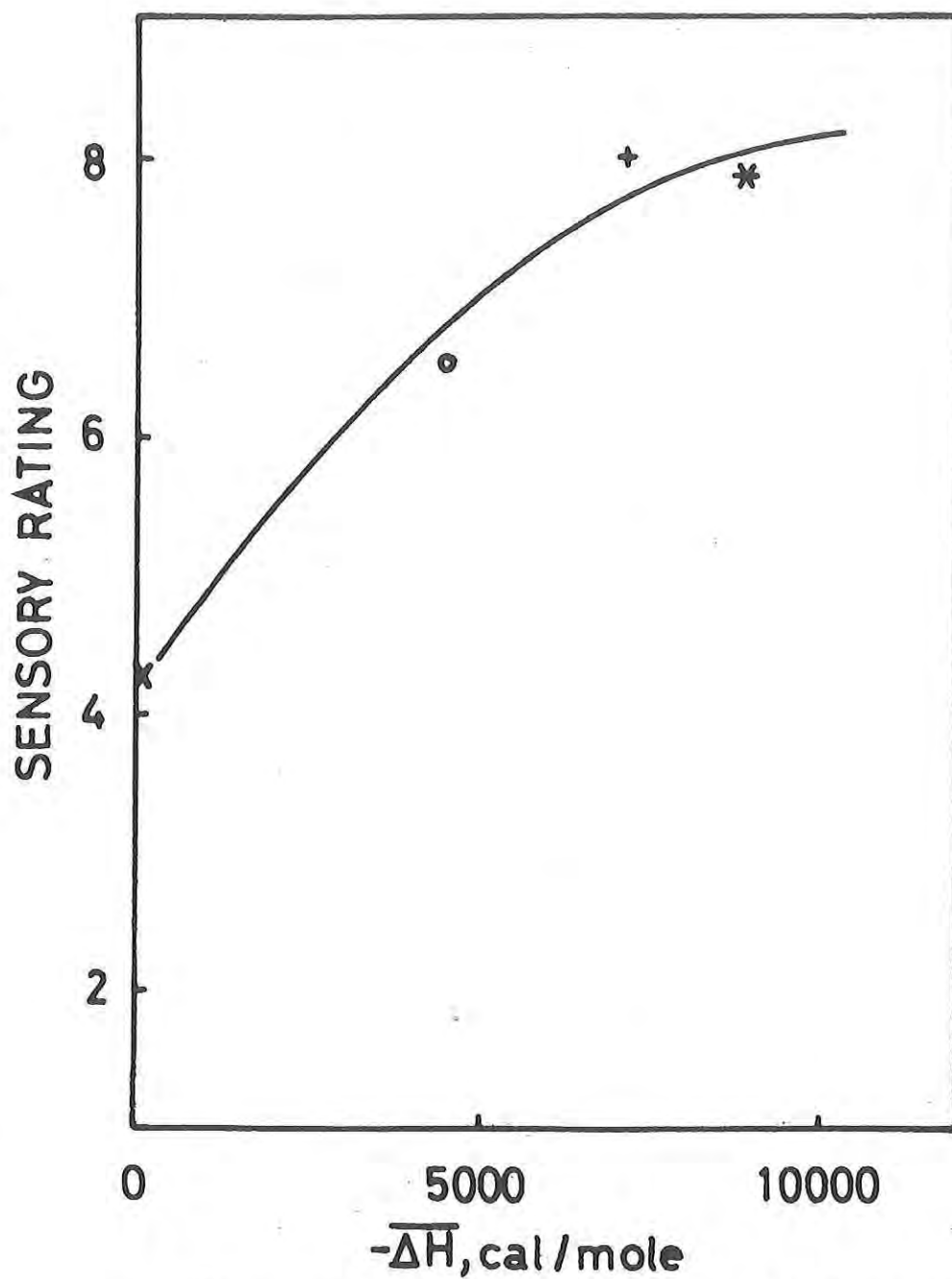


Fig. 58. Plot of sensory ratings for heat evolved in the mouth during mastication vs. values of % relative humidity in pre-cooked freeze-dried beef, S.I.K. III and IV.



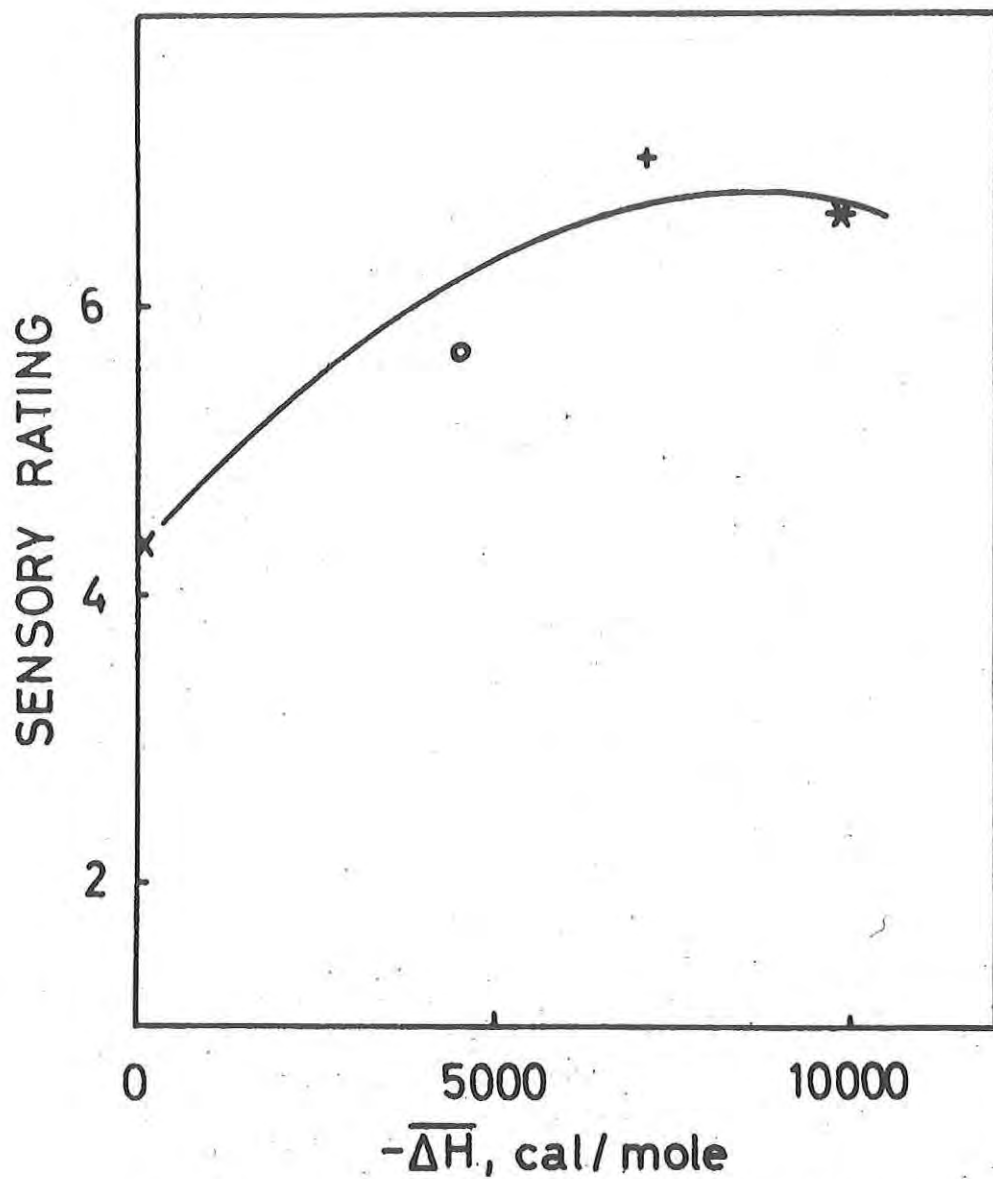
* = 0, + = 12, o = 23, x = 66 % R.H. at 20°C

Fig. 59. Plot of values of the partial molal enthalpy change vs. ratings for sensory hardness determined parallel to meat fibers before rehydration in the mouth, in pre-cooked freeze-dried beef, S.I.K. III.



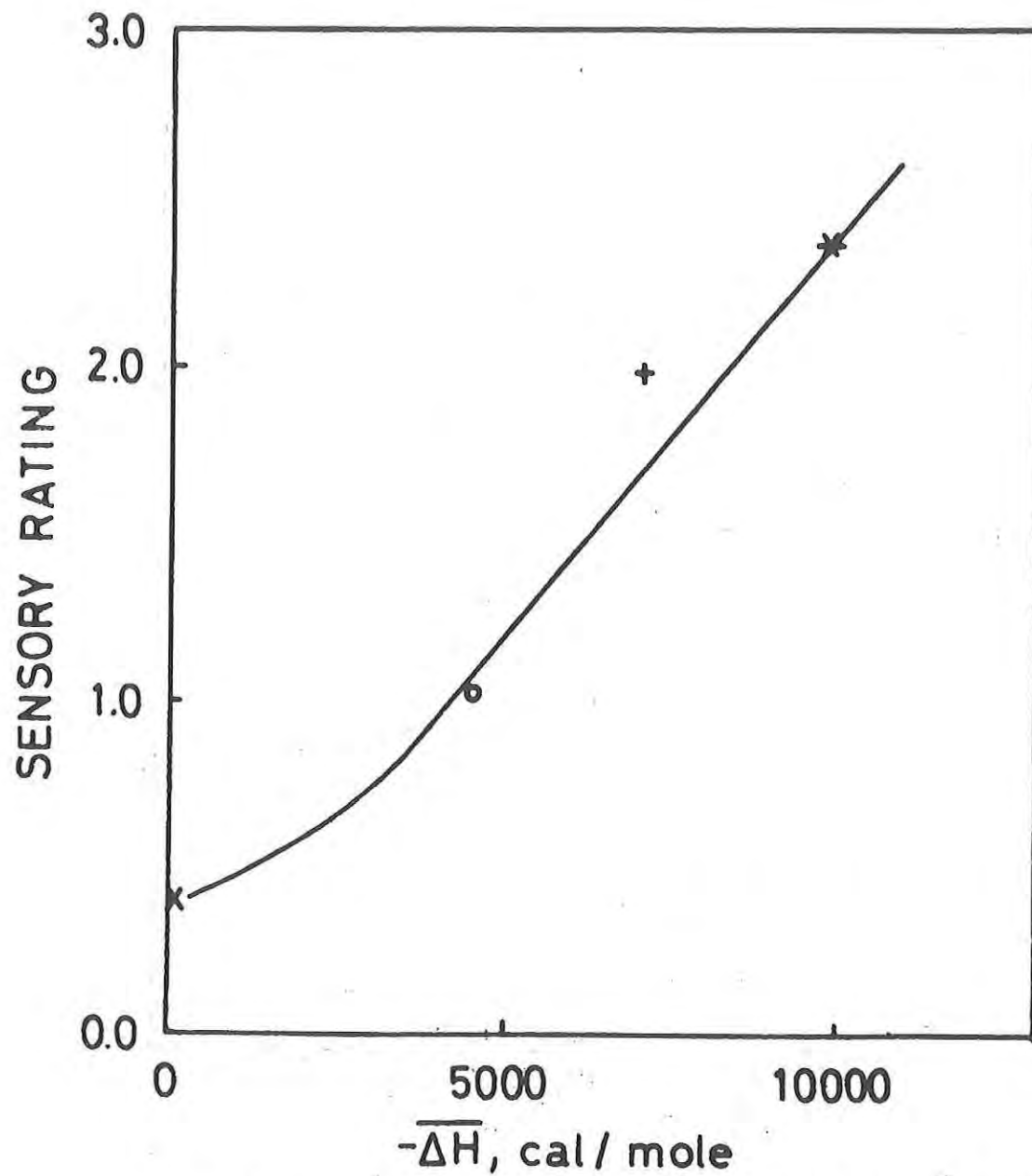
* = 0, + = 12, o = 23, x = 66 % R.H. at 20°C

Fig. 60. Plot of values of the partial molal enthalpy change vs. ratings for sensory hardness determined across meat fibers before rehydration in the mouth, in pre-cooked freeze-dried beef, S.I.K. III.



* = 0, + = 12, o = 23, x = 66 % R.H. at 20°C

Fig. 61. Plot of values of the partial molal enthalpy change vs. ratings for sensory tenderness determined after rehydration in the mouth, in pre-cooked freeze-dried beef, S.I.K. III.



* = 0, + = 12, o = 23, x = 66 % R.H. at 20°C

Fig. 62. Plot of values of the partial molal enthalpy change vs. sensory ratings of heat evolved in the mouth during mastication, in pre-cooked freeze-dried beef, S.I.K. III.

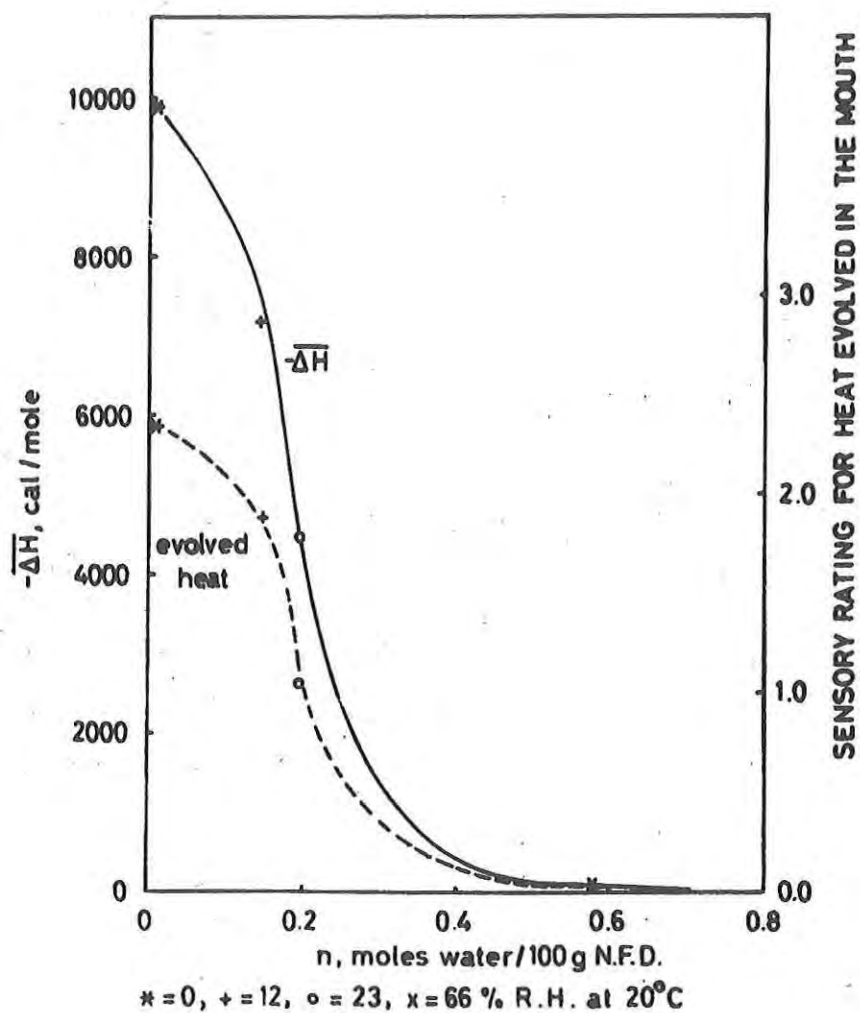
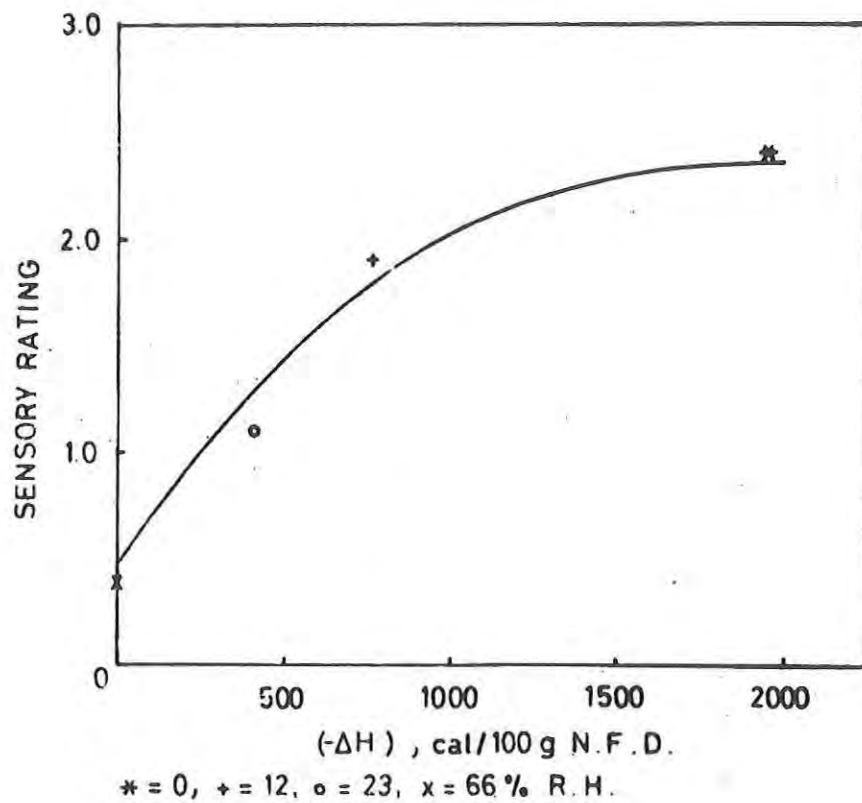


Fig. 63. Plot of values of the partial molal enthalpy change and of heat evolved in the mouth during mastication vs. equilibrium moisture in pre-cooked freeze-dried beef, S.I.K. III.



* = 0 + = 12 o = 23 x = 66 % R.H.

$(-\Delta H)$ = integral net ΔH at saturation - integral net ΔH at corresponding % R.H.

Fig. 64. Plot of values of the integral net enthalpy change (ΔH) vs. heat evolved in the mouth during mastication, in pre-cooked freeze-dried beef, S.I.K. III.

STATISTICAL ANALYSIS

Significance levels obtained in the Analysis of Variance for the instrumental measurements on the S.I.K. II sample series are presented in Table 37. (The data for the S.I.K. I, III and IV sample series had not been processed by the IBM Data Center at the time of the writing of this Report. The results for all sample series will be included in future publications derived from the data of this Report).

Instrument Force. In the dry food, the instrument force was different at the 0.1 % level of significance for the following variables: depth of penetration, humidity and position of muscle (left or right). Stated otherwise, these variables exercised a highly significant effect on the values obtained for hardness by the Masticometer. All interactions between these variables were not significant. The lack of significance for the depth-humidity interaction may indicate a uniform moisture distribution throughout the mass of the meat after the equilibration period.

In the rehydrated food, the instrument force was different at the 0.1 % level for the depth of penetration and the position of the muscle, and at the 5 % level of significance for the relative humidity. Compared with the results for the material in the dry state, this indicated that the relative humidity had a much greater effect on the force in the dry than in the rehydrated food. These results are in agreement with the plots in Figs. 24 and 25.

Cohesiveness. In the dry material, the cohesiveness value was different at the 0.1 % level of significance for the depth of penetration and for the relative humidity. Cohesiveness increases with depth, due to the fact that the binding forces which hold the elements together in the structure, offer greater resistance to penetration as the depth increases. The results on the humidity are interesting, since they indicate that these

binding forces become stronger with increasing moisture content. This gives support to my previous interpretation that higher moisture levels in the meat were associated with cross-linking reactions which toughened the meat.

In the rehydrated food, the results are similar to those obtained for the dry material, although the absolute magnitude of the F value (which is not shown in table 37) was smaller in the rehydrated than in the dry food.

Crushability index. In the dry meat, the crushability index was different at the 0.1 % level for the depth of penetration, at the 5 % level for the position of the muscle, and at the 1 % level of significance for the depth-position interaction. The highly significant effect of the depth of penetration on the crushability index can be explained by the fact that the non-recoverable texture parameter increased with depth as the material was crushed.

In the rehydrated food, only the depth of penetration had an effect (at the 0.1% level of significance) on the crushability index.

The lack of significance for the effect of humidity on the crushability index is in general agreement with the results which were presented in Tables 22 and 23. It is interesting to compare these results with those obtained on the NLABS special foods, Table 27. In this latter case, the relative humidity had an appreciable effect on the crushability index, and this was attributed to the high carbohydrate content of these special foods.

Sensory evaluation. The results of the Analysis of Variance for the sensory evaluation data are shown in Table 38. Of all variables, the humidity had the greatest effect on the different sensory parameters. This effect was significant at the 0.1 % level for hardness determined parallel to and across the meat fibers for all the S.I.K. sample series, and for tenderness determined after rehydration in the mouth for the S.I.K.

III. sample series. It was significant at the 1 % level in tenderness for the S.I.K. IV sample series. The effect of humidity on flavor was less pronounced but still evident. Of the interactions between variables, the humidity-judge (HxJ) interaction for hardness and tenderness was not significant for the S.I.K. III, and significant at the 1 % level for the S.I.K. IV sample series. In the latter case, it appears that different judges rated the samples in different ways for the different humidity conditions. It is possible that the series IV samples, due to the type of the animal used or other reasons, had certain peculiarities in their moisture sorption characteristics which affected the different judges in different ways. In spite of the significant HxJ interaction for the S.I.K. IV sample series, the effect of humidity on all sensory texture parameters, as stated above, was very pronounced.

The lack of significance, in most cases, for the interactions Humidity-Taste session, and Judge-Taste session indicated the consistency of the sensory testing by the judges, independent of session. This is also supported by the fact that no significant effect of the taste session on the different parameters was observed.

Table 37. Compilation of the significance levels obtained in the Analyses of Variance for the S.I.K.
II sample series. Instrumental measurements.

Source of variation	df	Force		C ⁽¹⁾		C ⁽²⁾	
		Dry	Rehydrated	Dry	Rehydrated	Dry	Rehydrated
<u>Main effects:</u>							
Between depths, D	3	+++	+++	+++	+++	+++	+++
" humidities H	3	+++	+	+++	+++	n.sign.	n.sign.
" positions P	1	+++	+++	n.sign.	n.sign.	+	n.sign.
<u>Interactions:</u>							
DxH	9	n.sign.	n.sign.	n.sign.	n.sign.	n.sign.	n.sign.
DxP	3	n.sign.	++	n.sign.	n.sign.	++	n.sign.
PxH	3	n.sign.	n.sign.	n.sign.	n.sign.	n.sign.	n.sign.
DxHxP	9	n.sign.	n.sign.	n.sign.	n.sign.	n.sign.	n.sign.

(1) Cohesiveness

(2) Crushability index

n.sign. = not significant

+ = significant at the 5% level

++ = significant at the 1% level

+++ = significant at the 0.1% level

Table 38. Compilation of the significance levels obtained in the Analyses of Variance for the S.I.K. III and IV sample series. Sensory evaluation

Source of variation	Hardness parallel to fibers		Hardness across fibers		Flavor		Tenderness	
	III	IV	III	IV	III	IV	III	IV
<u>Main effects:</u>								
between humidities H	+++	+++	+++	+++	+	++	+++	++
" judges J	++	n.sign	n.sign	n.sign	n.sign	n.sign	++	n.sign
" taste sessions T	n.sign	n.sign	n.sign	n.sign	n.sign	n.sign	n.sign	n.sign
<u>Interactions</u>								
H x J	n.sign	++	n.sign	++	++	++	n.sign	++
H x T	n.sign	n.sign	n.sign	n.sign	+	n.sign	n.sign	n.sign
J x T	n.sign	n.sign	n.sign	n.sign	++	n.sign	n.sign	n.sign

n.sign = not significant

+ = significance at the 5% level

++ = significance at the 1% level

+++ = significance at the 0.1% level

SUMMARY AND CONCLUSIONS

The effect of the moisture content-water vapor equilibrium on the textural parameters of special dehydrated foods which can be consumed in the dry state was investigated. These foods included: (a) samples of four series of pre-cooked freeze-dried beef, obtained from different animals and representing different cooking variables, which were processed by S.I.K., and (b) samples of five special dehydrated foods used in space feeding, which were supplied by NLABS. The foods were equilibrated to different moisture contents, by storing at 0, 12, 23 and 66% R.H., under reduced pressure, at 20°C for 5½ months. The moisture content on non-fat dry (N.F.D.) basis corresponding to the 0 and 66% R.H. respectively, was 0 and about 12% for most of the S.I.K. samples, and 0 and about 16% for most of the NLABS samples. At the end of the storage period, the textural characteristics of the S.I.K. samples were investigated in both the dry and rehydrated states, whereas the textural characteristics of the NLABS samples were investigated only in the dry state.

The mechanical texture parameters were determined by a new instrument, the S.I.K. Masticometer, which had been constructed prior to the Report. The instrument is a modification of the M.I.T. -G.F. Texturometer, and it provides (a) high sensitivity, (b) good reproducibility, and (c) the possibility of evaluating the textural properties of a food at different depths of penetration within the sample. Item (c) is accomplished by a graphical analysis of the recorded curve, which, when further applied, leads to the quantitative description of a texture profile within complete chewing cycles. A new parameter, the "crushability index", and associated concepts of crushing, elastic, and total texture behavior of the food, were applied to the study of the experimental samples, in addition to the already known texture parameters of hardness and cohesiveness.

The sensory texture parameters were evaluated on two series of the S.I.K. samples in the dry state, using a specially trained sensory panel.

At the time of writing of this Report, results were available from experiments on the NLABS samples, from experiments and Analyses of Variance on the S.I.K. II samples, and from experiments and Analyses of Variance for the sensory evaluation on the S.I.K. III and IV samples. These results may be summarized as follows:

Increasing relative humidity values from 0 to 66% caused an appreciable increase in hardness, and a relatively smaller increase in % cohesiveness of most samples. This was more evident on the samples in the dry than in the rehydrated state, and it occurred to a greater extent at relative humidities above the value of the B.E.T. monomolecular layer of water. (For reference reasons, it can be stated that the B.E.T. monomolecular layer of water value at 20°C in most samples corresponds to about 20% R.H., and 3.5% moisture on N.F.D. basis). The same conditions caused a substantial decrease of the crushability index of the NLABS samples, but in the majority of cases, they did not materially affect the crushability index of the S.I.K. samples. (The latter result should be considered with caution until the statistical analysis for the S.I.K. I, III and IV sample series is available).

These results, and the background information provided in the scientific literature, indicate that the toughening effect on the meat of increasing moisture content may be due to cross-linking, and possibly other chemical reactions, which occur to a much greater extent at values above the B.E.T. monomolecular layer of water.

A number of recommendations with regard to instrumentation of objective texture evaluation, and to possible means for the improvement of the textural quality of dehydrated special foods can be made.

With regard to the Masticometer, I suggest that the academic and/or industrial talent of the United States in the instrument field be recruited to consider: (a) effective use of other means,

besides strain-gauges, for the measurement of deformation and/or stress in foods, (b) possible improvements in the design of the instrument beam, in the location of the strain-gauges on the reed with reference to the beam, and in the location of the motor with reference to the other parts of the apparatus, (c) use of an automatic integrator for the calculation of surface area and of Masticometer parameters by reference to a correctly positioned base line, (d) possible improvements in the electronic circuitry, and (e) a mathematical analysis of the deformation of the sample as caused by the Masticometer punch movement.

I suggest that a ~~strong~~ effort be made for an in-house research capability in the texture area. This can be done only by (a) assigning the necessary spaces for qualified scientific staff, and (b) obtaining the required instrumentation. In the latter case, besides the Masticometer described in this Report, the Instron Tensile Tester (with appropriate accessories), and the recent model of the Kramer Shear-Press should be purchased for texture research on full-time basis.

With regard to possible methods for the improvement of the textural quality of dehydrated special foods, the recommendations presented here are based, as stated previously, on the experimental data for the NLABS samples, and on the results which were available at the time of writing of this Report. These recommendations may be subject to change when the results of the statistical analysis of the S.I.K. I, III and IV sample series are available.

In most foods, undesirable increase in hardness and cohesiveness which occurs upon storage, may be reduced by keeping the moisture content below the value of the B.E.T. monomolecular layer of water. Excessive crumbliness, which was evident in all the space foods of this Report, could be avoided by increasing the relative humidity to as high as 66%. However, since high % R.H. values, as mentioned above, cause also an increase of hardness and

cohesiveness, a workable "balance" or "target" could be struck by dehydrating the food to a moisture content corresponding to two B.E.T. monolayer values.

Since special space foods are novel items in the food field, research should be initiated in order to define the desirable textural parameters of each type of food, from both the instrumental and the sensory point of view. This will provide the background information for any further effort to improve the textural qualities of these foods.

In most cases, the effect of the hygroscopic equilibrium, at different points of the isotherm, on the textural properties of the food was exerted by a modification of the contribution of the elastic and the non-recoverable parameters to the total texture behavior of the food. It is possible that a substantial improvement of the textural quality can be achieved by a modification of those parameters through various means. This includes changes in the composition, pre-cooking and/or freeze dehydration variables, use of additives, and measures to prevent or reduce cross-linking reactions, especially amino-carbonyl and disulfide bond formation.

I suggest that many of the above mentioned aspects be studied first on model systems, where the composition and other variables can be accurately controlled. The information obtained on these systems can then be tested on natural foods. This may materially increase the capability of the food research program of the Army in a scientifically important, and logistically essential frontier of knowledge.

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13. ABSTRACT			
<p>Improved methodology for the objective evaluation of food texture was applied to the measurement of the textural parameters of special freeze-dried foods which were storage-equilibrated under different relative humidity conditions. The foods included pre-cooked freeze-dried beef, laminated freeze-dried products in the form of bite-size sandwiches, and compressed freeze-dried cubes, all of which were designed for consumption without prior rehydration. The results showed that increasing relative humidity from zero to 66% caused an increase in hardness and cohesiveness, and, in certain foods, a decrease in brittleness (crushability index). This was more pronounced at equilibrium points above the B.E.T. value for a monomolecular layer of water. Plots of physico-chemical parameters of water vapor sorption versus rheological properties at different points of the moisture sorption isotherm were examined. The selection of certain "target" relative humidity values to minimize undesirable changes in storage, and promising avenues for continued work on the textural quality and acceptability of special freeze-dried foods are discussed.</p>			

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Measurements	8					
Texture	9		7			
Parameters	9		7			
Dehydrated Foods	9		9			
Freeze-dried Foods	9		9			
Humidity			6			
Relative Humidity			6			
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